

ANISOTROPIC MECHANICAL PROPERTIES OF TEXTURED QUARTZ PORCELAIN

IGOR ŠTUBŇA, ALŽBETA LINTNEROVÁ, LIBOR VOZÁR

*Department of Physics, Constantine the Philosopher University,
A. Hlinku 1, 949 01 Nitra, Slovak Republic*

E-mail: istubna@ukf.sk

Submitted November 19, 2007; accepted February 25, 2008

Keywords: Porcelain electroceramics, Technological texture, Young's modulus, Flexural strength

Textured samples prepared from 50 % kaolin, 25 % feldspar and 25 % quartz were fired at selected temperatures from 200 to 1320°C. Young's modulus and flexural strength were measured. The results showed the dependence of mechanical properties on the orientation of the kaolinite crystals in the sample. Young's modulus is higher along the basal planes of the kaolinite crystals. Flexural strength had lower values if a force affected the sample collinearly with basal planes of the kaolinite crystals. Both mechanical strength and Young's modulus showed that the texture effects persisted even after firing.

INTRODUCTION

It is often assumed that due to their polycrystalline structure, ceramic materials are isotropic. But anisometric crystals or particles, which are part of the dry ceramic powder or the water ceramic slip, can be spatially oriented if an applicable method is used. For example, spray-deposition leads to a preferential arrangement of particles parallel to the substrate surface [1]. Similarly, a cold pressed and sintered body seems to have a preferred orientation with the particles (crystals) lying nearly parallel to the direction of the pressing force [2]. A similar effect occurs during hot-deformation processing (moderate texture) or templating with aligned platelets (stronger texture) [3] and hot-pressing. Crystal rotation during pressing and preferred crystal growth of plate-shaped crystals during annealing may also contribute to the texture [4]. Another method is vibration of a container with anisometric powder particles, where the particles are affected by the gravitational force, when particles were exposed to vibration and preferred orientation of the basal planes in the horizontal level was obtained [5, 6]. The texture persists in the green electroceramic blank obtained from the vacuum extruder. This was shown by radiointroscopy based on penetrating plane-polarized electro-magnetic waves through the slab sample cut from the blank. Well-ordered texture can be identified in the outer layer of that blank [7].

The influence of the texture on the mechanical strength of ceramics is typically considered to be harmful. Adjacent volume blocks with a different texture

may cause cracking on block boundaries during drying or firing. The cause of that is anisotropy of thermal expansion of the volumes with the different orientation of anisometric crystals. The acoustic emission method was employed to detect microcracking attributable to the thermal expansion anisotropy. Acoustic signals were registered during cooling and a direct correlation between microcracking and anisotropy was observed [8, 9]. Microcracks preferentially form at grain boundaries and propagate either in the bulk or along grain boundaries, depending on the toughness of the boundaries relative to the grain interiors [10]. The fracture toughness of the samples measured parallel to the pressing direction was higher than that perpendicularly measured to the pressing direction [4].

Anisometric platelike kaolinite crystals represent the most significant part (up to 50 %) of the raw material mixture used in the electroceramic plants for insulator production. Forming of a wet ceramic mass by using the vacuum extruder aligns the anisometric crystals into a texture with a different local degree of the order. Measurements of the material show the anisotropy of mechanical strength [11], velocity of sound propagation [12], thermal diffusivity [13], and electrical properties [7, 14, 15]. The texture affects the dimensions of a wet sample during its drying [16] and the sample dilatation during heating [17].

The highest degree of texture is created in the surface layer of the blank as a result of friction between the wet ceramic mass and the mouth of the vacuum extruder.

A reliable confirmation of the texture in the surface layer of the green blank for quartz porcelain may be done using XRD analysis. This method was used in [13, 17] for differentiating the texture of green porcelain ceramic samples obtained by vacuum extruding. A significant increase of the intensities of the kaolinite diffraction peaks (0001) and (0002) was observed for samples cut from the cylindrical blank in the radial direction compared to samples cut in the axial direction. XRD analysis confirmed that basal planes of thin pseudo-hexagonal kaolinite crystals are predominantly perpendicular to the radius of the blank [17]. It was also observed that feldspar crystals are partially oriented and the quartz crystals are randomly oriented [15]. Evolution of mullite texture on firing tape-cast kaolin bodies indicated that the texture preserves during heating [18].

The objective of this paper is to illustrate how technological texture influences the mechanical strength and Young's modulus of quartz electroporcelain.

EXPERIMENTAL

Electroporcelain samples were prepared in the following manner: a mixture of kaolin (50 wt.%), quartz (25 wt.%), and feldspar (25 wt.%), employed for high voltage insulator production, was wet ground and partially dewatered by pressure filtering. From the resulting plastic mass, a blank with diameter of 200 mm was extruded using a vacuum extruder. After reaching the equilibrium moisture (with about 1 wt.% of the physically bound water), the samples were cut from the blank. The method for cutting the samples from the surface layer is shown in Figure 1. The dashed lines show the prevailing orientation of the kaolinite crystals in the samples.

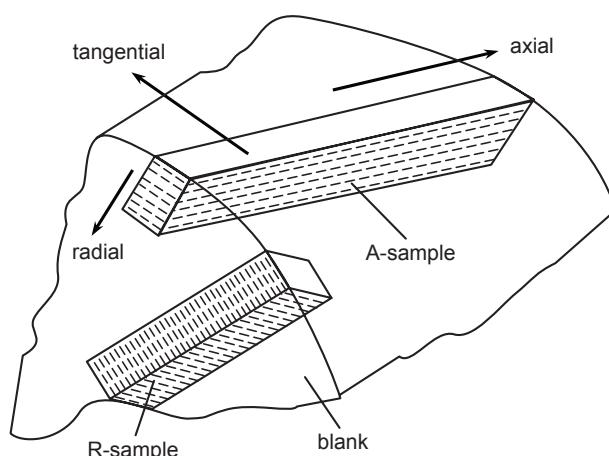


Figure 1. Samples and directions of the texture. Dashed lines indicate orientation of the platelike kaolinite crystals.

The technological texture was detected using the XRD analysis with CuK α radiation applied on the green compact samples cut according to Figure 1.

Young's modulus was measured by the impulse ultrasound method on the samples with dimensions 17×17×17 mm. The ultrasound wave was directed perpendicularly to the basal planes of the kaolinite crystals (radial direction) or collinearly with these planes (axial or tangential direction). Five samples were fired at selected temperatures from the interval of 200 to 1320°C (the temperatures are showed in Figure 2) using a heating rate 5°C/min, 10 min holding the temperature, and then free cooling down with a furnace. Young's modulus was calculated from the sound velocity c and volume mass ρ by the formula $E = c^2 \rho$. The volume mass was calculated from the weight and dimensions of the sample.

The flexural strength was measured by the three-point-bending test with the support span of 60 mm. Dimensions of the samples were 9×9×70 mm. Sets of green or fired (1320°C, 10 min) samples of both textures were measured at the room temperature. The resultant values of the flexural strength were processed by Weibull's statistics.

RESULTS AND DISCUSSION

XRD analysis

The technological texture of the samples was confirmed by XRD analysis. A significant enhancement of K(0001) and K(0002) diffraction peaks of R-sample compared with the A-sample, affirmed that the basal planes of the kaolinite platelet crystals are predominantly perpendicular to the radius of the blank, see Table 1. These results are consistent with results obtained in [13, 14, 15].

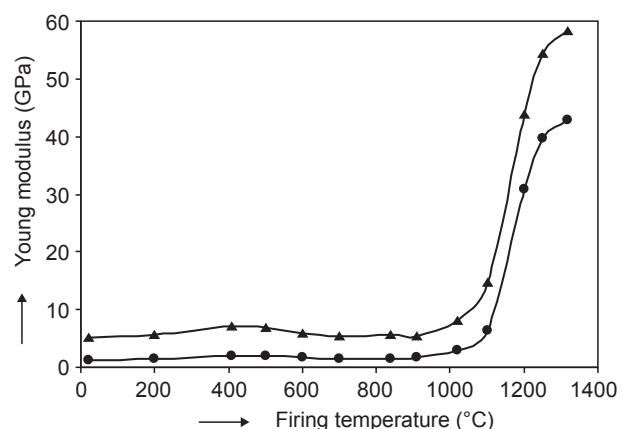


Figure 2. Dependence of the Young's modulus on firing temperature for radial direction (●) and axial direction (▲)

Table 1. Intensity of the diffraction peaks (in arbitrary units) of the quartz electroporcelain samples.

Diffraction peak intensity			
Axial direction		Radial direction	
K(0001)	K(0002)	K(0001)	K(0002)
100	58	348	236

Young's modulus

Results of the ultrasound measurements of the Young's modulus with ultrasound wave passing through the sample along the basal planes of the kaolinite crystals (axial or tangential direction) and perpendicularly on the basal planes (radial direction) are displayed in Figure 2. Only graphs for axial and radial directions appear in Figure 2, because the results for axial and tangential directions were close. This follows from the similar orientation of the anisometric crystals in these directions. The results indicated a strong dependence of the Young's modulus on the texture. The results also show that differences between Young's moduli in radial and axial/tangential directions were preserved even after firing at the temperature of 1320°C. The results reflect the main processes in ceramic material - dehydroxylation accompanied by slight decrease of the Young's modulus [19] and rapid solid-phase sintering at the temperatures above 950°C.

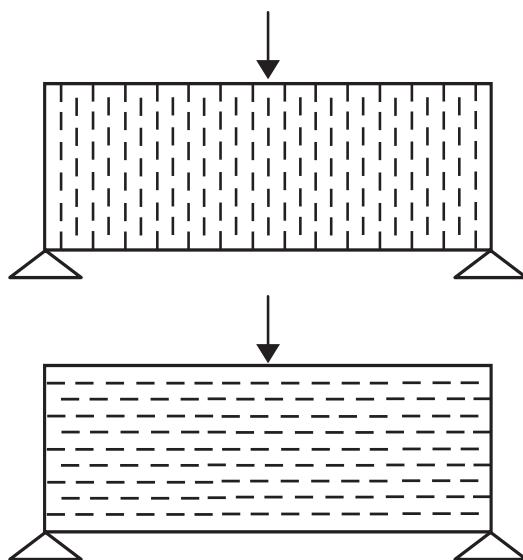


Figure 3. Three-point bending the R-samples with radial texture (upper picture) and A-samples with axial texture (lower picture). Dashed lines indicate positioning of the platelike kaolinite crystals.

Mechanical strength

The results of the Weibull's statistics used for processing the mechanical strength (measured by the three-point bending, see Figure 3) of the green and fired samples are shown in Figures 4-7.

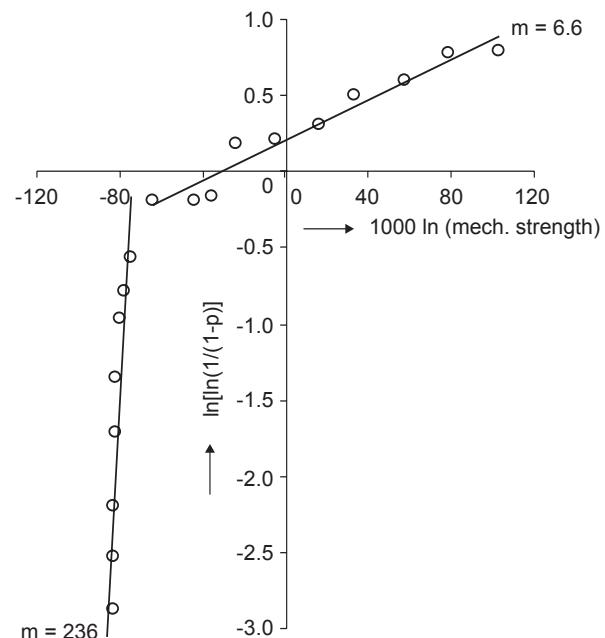


Figure 4. Results of the Weibull's statistics for mechanical strength (MPa) of the green R-samples. Weibull's moduli are written beside the linear approximations.

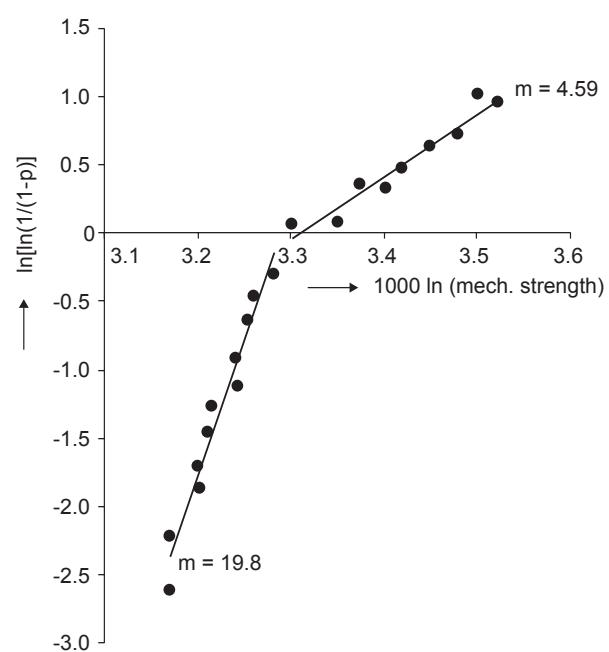


Figure 5. Results of the Weibull's statistics for mechanical strength (MPa) of the fired R-samples.

Obviously, there are two types of texture of R-samples which can be identified in Figures 4-5. The first type has lower mechanical strength and high Weibull's modulus that provides evidence of the common dominant source of the cracks. The second type of the texture has higher mechanical strength and low Weibull's modulus which means a large influence of sample size on the strength values. The fired R-samples preserve this character of the mechanical strength. However the Weibull's moduli are lower and mechanical strengths are much higher.

Similarly, the A-samples also decrease their Weibull's modulus after the firing; see Figures 6-7. However, in this mode of the mechanical loading, the influence of the defects in the structure is not as dominant as in the R-samples. Thus, measuring the mechanical strength before and after firing (1320°C) also supports the fact, that the mechanical properties are sensitive to texture, and that axial texture clearly implies higher mechanical parameters. A comparison between the mechanical strengths of A-samples and R-samples is shown in Table 2. It can be assumed that the mechanical strength of the non-textured sample is between the values in the A-samples and R-samples.

Figures 4-7 show different mechanical behavior of the A-samples and R-samples. It was found that the texture of the extruded blank is complex and consists of two partial textures - helical and circular. The circular texture appears mostly on the surface of the blank, while the interior of the blank has a combined texture [20]. From this point of view, the A-samples and R-samples have different textures. The texture of the A-sample (which is cut from the surface of the blank) is mostly circular (see Figure 1) and relatively homogeneous. The texture of the R-sample is combined because it is cut radially from the blank and its center (which is a place of the fracture) is 35 mm under the surface (see Figure 1). The results seen in Figures 4-5 reflect the double texture of the R-samples.

It is important to note that the numerical values shown in Table 2 and in Figure 2 and Figures 4-7 should not be considered as generally valid. If the samples were cut from another location of the blank or another vacuum extruder used, the values would be different than presented in this article. This was also found in [21], where the texture was determined indirectly by the contraction of the small samples after drying and firing, and cut from different vacuum extruded blanks.

Both Young's modulus and mechanical strength confirmed that the texture is sustained even after firing at 1320°C . This fact can be explained by mullite crystals which come from the kaolinite crystals [22, 23]. XRD analysis of the samples cut from the extruded blank before and after firing confirmed the same orientation of the mullite crystals as original kaolinite crystals [24].

Figure 6. Results of the Weibull's statistics for mechanical strength (MPa) of the green A-samples.

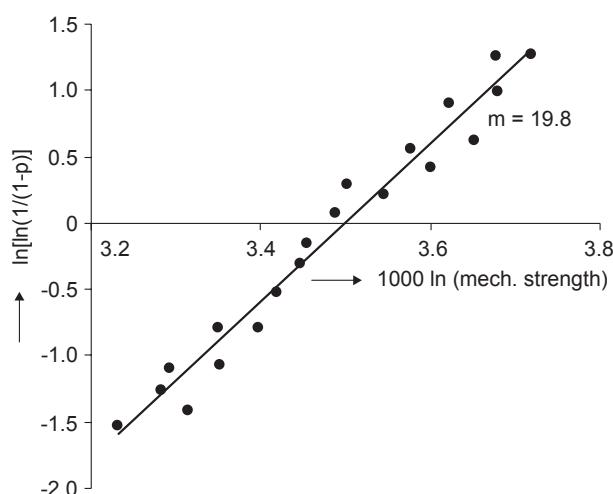


Figure 7. Results of the Weibull's statistics for mechanical strength (MPa) of the fired A-samples.

Table 2. Mechanical strength (MPa).

A-samples		R-samples	
green	fired	green	fired
1.02 ± 0.06	32.82 ± 4.93	0.98 ± 0.06	27.64 ± 3.27

CONCLUSION

The tests showed dependence of the mechanical properties on the orientation of the kaolinite crystals in the sample. Young's modulus is higher along the basal planes of the kaolinite crystals. Flexural strength has lower values if a force affects the sample collinearly with basal planes of the crystals. Young's modulus and flexural strength preserve anisotropy during the all the firing.

Acknowledgement

This work was supported by the VEGA grant 1/3179/06. The authors thank the ceramic plant PPC Čab for providing green ceramic samples.

References

1. Wanner A., Lutz, E.: J.Am.Ceram.Soc. 81, 2706 (1998).
2. Boatner L. A., Bolduo J. L., Abraham M. M.: J.Am.Ceram. Soc. 73, 2333 (1990).
3. Hall P. W., Swiney J. S., Kovar D.: J.Am.Ceram.Soc. 84, 1514 (2001).
4. Kim W., Kim Y. W., Chen D. H.: J.Am.Ceram.Soc. 81, 1669 (1998).
5. Minárik S., Kozík T., Kalužný J., Labaš, V.: Proc. conf. CO-MAT-TECH 1996, p.95, TF STU Trnava 1996.
6. Kozík T., Kalužný J., Minárik S., Labaš V.: Interceram 48, 104 (1999).
7. Beljanin A. N., Rudakov V. N.: Izv. Vuzov-Defektoskopija 4, 1 (1968).
8. Srikanth V. et al.: J.Am.Ceram.Soc. 74, 365 (1990).
9. Srikanth V., Subbarao E. C., Rao, G. V.: Ceramics International 18, 251 (1992).
10. Sridhar N., Yang W., Srolovitz D.: J.Am.Ceram.Soc. 77, 1123 (1994).
11. Štubňa I., Kozík T.: Sklář a keramik 33, 80 (1983).
12. Kalužná M., Kaprálik I., Štubňa I.: Sklář a keramik 37, 291 (1987).
13. Kalužná M., Vozár L., Gembarovič J., Kaprálik I.: Ceramics-Silikaty 36, 15 (1992).
14. Trnovcová V., Furár I., Hanic F.: Physics and Chemistry of Solids 68, 1135 (2007).
15. Trnovcová V., Kalužná M., Furár I., Hanic F., Filanová J.: Proc. Conf. CO-MAT-TECH 2005, p. 1230, MTF STU Trnava 2005.
16. Vycudilík P.: Sklář a keramik 28, 50 (1978).
17. Štubňa I., Valovič Š.: Industrial Ceramics 24, 121 (2004).
18. Chen. Ch. Y., Tuan W. H.: J. Am. Ceram. Soc. 85, 1121 (2002).
19. Štubňa I., Trník A.: Ceramics-Silikaty 51, 102 (2007).
20. Iqbal, Y. - Lee, W.E.: J.Am.Ceram.Soc. 83, 1321 (2000).
21. Kozík T., Labaš V., Olvecký Š.: Silika 15, 131 (2005).
22. Hofmann M., Danheim H.: Keram. Zeitschrift 39, 86 (1987).
23. Gaultieri, A. F.: J.Am.Ceram.Soc. 90, 1222 (2007).
24. Sharov V. I., Moroz I. C.: Steklo i keramika, N7, 24 (1986).

ANIZOTROPICKÉ MECHANICKÉ VLASTNOSTI TEXTUROVANÉHO KREMIČITÉHO PORCELÁNU

IGOR ŠTUBŇA, ALŽBETA LINTNEROVÁ,
LIBOR VOZÁR

Katedra fyziky, Univerzita Konštantína Filozofa,
A. Hlinku 1, 949 01 Nitra, Slovensko

Texturované vzorky pripravené zo zmesi 50 % kaolínu, 25 % živcov and 25 % kremeňa boli vyrezané z valcového výlisku (obr. 1) a vypaľované pri vybraných teplotách od 200 do 1320°C. Textúra bola potvrdená rtg. difraktometriou (Table 1). Bol meraný Youngov modul pružnosti ultrazvukovou metódou (obr. 2) a mechanická pevnosť trojbodovým ohybom v dvoch smeroch orientácie kaolinitových kryštálov (obr. 3). Výsledky merania mechanickej pevnosti boli spracované Weibullovou štatistikou (obr. 4, 6 pre súrové vzorky a obr. 5, 7 pre vypálené vzorky). Výsledky ukázali závislosť týchto veličín na orientácii kaolinitových kryštálov vo vzorke. Youngov modul je vyšší v smere rovnobežnom s bazálnymi rovinami kryštálov. Mechanická pevnosť v ohybe má menšie hodnoty, ak zaťažujúca sila pôsobí rovnobežne s bazálnymi rovinami (Table 2). Anizotropia mechanickej pevnosti a Youngovho modulu sa zachovala aj po vysokoteplotnom výpale.