THE INFLUENCE OF ILLITE-KAOLINITE CLAYS' MINERAL CONTENT ON THE PRODUCTS' SHRINKAGE DURING DRYING AND FIRING

VPLIV VSEBNOSTI GLIN ILINIT-KAOLINIT NA KRČENJE PRI SUŠENJU IN ŽGANJU

Milun Krgović¹, Nada Marstijepović¹, Mileta Ivanović¹, Radomir Zejak², Miloš Knežević², Snežana Đurković¹

¹University of Montenegro, Faculty of Metallurgy and Technology, Cetinjski put bb, 81000 Podgorica, Montenegro ²University of Montenegro, Faculty of Civil Engineering, Cetinjski put bb, 81000 Podgorica, Montenegro milun@cg.ac.yu

Prejem rokopisa – received: 2007-01-19; sprejem za objavo – accepted for publication: 2007-03-18

In this paper an investigation of the influence of the mineral content of illite-kaolinite clays on the products' shrinkage during drying and firing is presented. Under the same conditions for preparing raw materials and ceramic mass, as well as under the same firing regime, the products' shrinkage during drying and firing is mostly influenced by the amounts of quartz and the illite and kaolinite clay minerals.

Keywords: clay, linear shrinkage, total shrinkage, sintering, porosity

Predstavljena je raziskava vpliva vsebnosti glin ilinit-kaolinit na krčenje pri sušenju in žganju. Pri enakih pogojih priprave surovega materiala in keramike in enakemu režimu žganja na krčenje najbolj vpliva razmerje med kremenom ter glino ilinit-kaolinit.

Ključne besede: glina, linearno krčenje, skupno krčenje, sintranje, poroznost

1 INTRODUCTION

The illite-kaolinite clays differ significantly in mineral composition from illite, kaolinite, quartz, feldspar, Fe₂O₃ and CaCO₃ ^{1,2}. The presented raw material composition, depending on the sintering temperature, produces solid-state reactions, polymorphic transformations of quartz and liquid-phase formation³. Besides the sintering temperature of the ceramic mass, the raw material mineral content also has an important role for the relations between the microstructure constituents⁴. The appearance of the liquid phase accelerates the solid-state reactions⁵. The mineral content of illite-kaolinite clays, besides other factors, determines the formation of new crystal phases during the sintering process, and the polymorphic transformations of quartz caused by volume changes⁶.

During the drying of shaped ceramic products, the simultaneous transfer of mass and heat in the homogeneous polydispersive system material with water occurs⁷. The water is transported from the internal area of the material through capillaries to the surface of the products, where it evaporates. The process depends on the diffusion and evaporation rates. The difference in the water content on the surface and in the inside layers of the material enables humidity to transfer continuously from the inside to the surface by diffusion⁸. The volume changes during the drying induce internal stresses.

2 EXPERIMENTAL

Two types of illite-kaolinite clays were used for the preparation of samples (the clays are marked "PV" and "BP"). The samples were formed by plastic shaping in a mould of parallelepiped shape with dimensions 7.7 cm \times 3.9 cm \times 1.6 cm and marked with the numbers 1, 2, 3, 4, 5 ... 15.

The clays' analyses consisted of a determination of the mineral content with x-ray analysis, as well as chemical and granulometric analyses with a determination of the particle size distribution. The grain shape was determined microscopically. The linear and volume shrinkage of the samples during the drying in air to a constant mass and during drying in a dryer at 110 °C were determined too.

The samples were fired at 800 °C, 900 °C, 1000 °C, 1100 °C and 1200 °C. The total porosity, the mineral content by x-ray analysis, and the microscopic analyses were assessed for the sintered samples.

3 RESULTS AND DISCUSSION

On the basis of the mineral and chemical compositions shown in **Figure 1**, **Figure 2** and **Table 1**, it was concluded that the investigated specimens were illite-kaolinite clay types containing α -quartz, carbonate and Fe₂O₃. In the "BP" clay the content of α -quartz was

Table 1: Chemical	composition of clay
Tabela 1: Kemična	a sestava gline

Oxides	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	Ig. loss
Clay "BP" Percentage by weight, <i>w</i> /%	72.68	5.70	10.98	0.48	0.70	0,31	1.12	_	8.03
Clay "PV" Percentage by weight, <i>w</i> /%	71	5.51	10.55	1.42	0.62	0.45	1.86	0.25	8.34

lower and there was a larger content of illite and kaolinite. The granulometric analysis shows a higher average content of "PV" clay grain (**Table 2**), although the pulverization of the clays was performed under the same conditions. This is due to the difference in mineral content, particularly the higher percentage of quartz in the "PV" clay.

The higher content of α -quartz in the "PV" clay and the higher average content of grains explain the lower average value of the volume shrinkage for the 15 samples during the air drying to a constant mass (Figure 3, Figure 4 and Table 3). The results of the volume shrinkage, as well as the average value for 15 samples during drying in a dryer to a constant mass, show higher values of the volume shrinkage for the samples based on "PV" clay (Figure 5, Figure 6 and Table 4). The higher the content of α -quartz, the lower the content of clay minerals and the higher average value of the grains in this clay enable the easier transport of water from the internal area of the samples through the capillaries to the surface at a drying temperature of 110 °C. The different values of the volume shrinkage of the samples during drying are the consequence of an unequal pressure during plastic shaping in a mould.

 Table 2: Average grains value of the investigated clay types

 Tabela 2: Povprečna velikost zrn v preiskanih glinah

Clay type	Average grains value (MV), d/µm
"BP"	17
"PV"	27

Table 3: Average values of volume shrinkage during drying in air to the constant mass

Tabela 3: Povprečno volumensko krčenje pri sušenju na zraku do konstantne mase

Clay type	Average values of volume shrinkage (for 15 samples) /%
"BP"	18.4
"PV"	15.8

Table 4: Average values of volume shrinkage during drying in dryer to the constant mass

Tabela 4: Povprečno volumensko krčenje pri sušenju v sušilniku do konstantne mase

Clay type	Average values of volume shrinkage (for 15 samples) /%
"BP"	0.98
"PV"	1.65

The volume shrinkage during firing is smaller for the samples based on the "PV" clay (**Figure 7**). The X-ray



Figure 1: X-ray diffractogram of "PV" clay Slika 1: Rentgenski difraktogram "PV"-gline



Figure 2: X-ray diffractogram of "BP" clay

Slika 2: Rentgenski difraktogram "BP"-gline



Figure 3: Volume shrinkage during drying to a constant mass in air ("PV" clay)

Slika 3: Volumensko krčenje pri sušenju do konstantne mase na zraku ("PV"-glina)

Materiali in tehnologije / Materials and technology 41 (2007) 4, 189-192



Figure 4: Volume shrinkage during drying to a constant mass in air ("BP" clay)

Slika 4: Volumensko krčenje pri sušenju do konstantne mase na zraku ("BP"-glina)



Figure 5: Volume shrinkage during drying to a constant mass in dryer ("PV" clay)

Slika 5: Volumensko krčenje pri sušenju do konstantne mase v sušilniku ("PV"-glina)



Figure 6: Volume shrinkage during drying to a constant mass in dryer ("BP" clay)

Slika 6: Volumensko krčenje pri sušenju do konstantne mase v sušilniku ("BP"-glina)



Figure 7: Volume shrinkage during sintering **Slika 7:** Volumensko krčenje pri žganju



Figure 8: X-ray diffractogram of sintered product ("BP" clay, T = 1200 °C)

Slika 8: Rentgenski difraktogram za sintrani produkt ("BP"-glina, T = 1200 °C)



Figure 9: X-ray diffractogram of sintered product ("PV" clay, T = 1200 °C)

Slika 9: Rentgenski difraktogram za sintrani produkt ("PV"-glina, T = 1200 °C)

M. M. KRGOVIĆ ET AL.: THE INFLUENCE OF ILLITE-KAOLINITE CLAYS' MINERAL CONTENT ...



Figure 10: Microstructure of sintered product ("PV" clay, T = 1200 °C)

Slika10: Mikrostruktura sintranega produkta ("PV"-glina, T = 1200 °C)

analysis of the sintered products shows a higher content of the minerals sillimanite, $Al_2O_3 \cdot SiO_2$, and mullite, $3Al_2O_3 \cdot 2SiO_2$, formed during the sintering of the samples based on "BP" clay (**Figure 8**). The higher content of the α -quartz in the "PV" clay and the higher total porosity explain the lower volume shrinkage during sintering, when the quartz is partly transformed into tridimite (**Figure 9**). The microstructures of the sintered products are shown in **Figures 10 and 11**.

4 CONCLUSION

The investigation of the influence of illite-kaolinite clays' mineral content on the linear and volume shrinkage during drying and firing shows that the shrinkage depends on the following factors:

• Using the same conditions for the preparation of raw materials and ceramic mass during drying in air and in a dryer to a constant mass, the most important factor is the influence of the relation between the quartz and clay's minerals content



Figure 11: Microstructure of sintered product ("BP" clay, T = 1200 $^{\circ}C)$

Slika 11: Mikrostruktura sintranega produkta ("BP"-glina, T = 1200 $^{\circ}\mathrm{C})$

• the values of the volume and linear shrinkage of samples during the firing process depend also on the relation between the quartz and the clay's minerals content. This influence is particularly strong at temperatures above 1000 °C.

5 REFERENCES

- ¹M. Tecilazić-Stevanović, *Principles of Ceramic Technology*, Faculty of Technology and Metallurgy, University of Belgrade, Belgrade (1990)
- ² R. E. Grim, *Clay Mineralogy*, New York: McGraw-Hill Book Co., (1983)
- ³ M. M. Krgović, N. Z. Blagojević, Ž. K. Jaćimović, R. Zejak, *Res. J. Chem. Environ.* 8 (**2004**) 4, 73–76
- ⁴ J. Griffiths, Ind. Min., 272 (1990), 35-40
- ⁵M. M. Krgović, Z. K. Jaćimović, R. Zejak, *Tile & Brick Int.* 17 (**2001**) 3, 178–181
- ⁶ M. Krgović, M. Ivanović, N. Z. Blagojević, Ž. Jaćimović, R. Zejak, M. Knežević, *Interceram*, 55 (2006) 2, 104–106
- ⁷ Lj. Kostić-Gvozdenović, R. Ninković, *Inorganic Technology*, Faculty of Technology and Metallurgy, University of Belgrade, Belgrade (1997)
- ⁸ B. Živanović, R. Vasić, O. Janjić, *Ceramic Tiles*, Institute of Materials in Serbia, Belgrade, (1985)