Characterization of phase transformation and thermal behavior of Sedlecky Kaolin

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In the case of ceramics, the used sintering method greatly influences the properties of the product [1–3], so it is important to know the effect of temperature. In this research, the authors have examined how the Sedlecky ml kaolin and alumina powder mixture behave under heating using a MOM Derivatograph-C and a CamarElettronica heating microscope [4]. From the powder mixture ceramic specimens were also made to determine how the volume, weight, and phases are changing using different sintering temperatures.

For the tests, kaolin and a mixture of kaolin and 10 m % alumina was milled in Retsch PM 400 planetary ball mill for 20 min at 150 rpm. The results of the thermo-analytical test of the kaolin-alumina mixture are shown in Fig. 1. From the achieved curves can be distinguished between drying 1, thermal degradation of kaolin 2-3 (conversion to

metakaolin), formation of mullite 4 and sintering point 5 (where the height of the sample compared to the original is 95 %).

Specimens were made from the mixtures with the uniaxially pressing method using a 100 kN mechanical pull-press machine. The pressed specimens were sintered in an electrical chamber kiln using different maximum kiln temperature and were kept at this temperature for 3 hours (Fig. 2).

The maximum temperature for sintering was chosen based on the $\text{SiO}_2\text{-Al}_2\text{O}_3$ phase diagram [5], waiting for the following phase transitions: 450 °C — kaolinite-metakaolinite; 575 °C — α -quartz- β -quartz; 775 °C — andalusite-sillimanite; 870 °C — β -quartz-tridymite; 1100 °C — metakaolinite-mullite transitions. As the sintering temperature increases, the color of the specimens changes continuously. If the sintering temperature is above 1000 °C the specimens became white. The change in color may indicate that the expected phase transitions have occurred.

Fig. 1. Thermo-analytical curves of kaolin and alumina mixture

Fig. 2. The specimens sintered at 450, 575, 775, 870, 1100 °C

Fig. 3. The changing of volume and weight under different temperature

Phase ratio for samples sintered at 1100 °C

Sign of the mixture		K	KA
Phase content, m %	Amorph	61.4	47
	Crystalline	38.6	53
	mullite	91.45	63.13
	tridymite	8.55	5.97
	corundum	-	30.9

Sintering at 450 °C increases the volume of specimens while decreasing their mass. At 575 °C, a weight loss of more than 10 % is observed, which is because the kaolinite-metakaolinite conversion is complete. The kaolinite mineral loses the crystalline water content (kaolinite mineral composition: 39.52 m % Al_2O_3 , 46.52 m % SiO_2 , 13.96 m % H_2O). The change in mass from 575 °C was already slightly influenced by the added Al_2O_3 content. The initial volume of specimens and the volume of specimens sintered at 1250 °C were approximately the same for both mixtures (Fig. 3).

The mineral composition was not significantly affected by the addition of Al_2O_3 and is present throughout the corundum phase in the samples due to the low sintering temperatures. During the sintering at 1100 °C, the mullite phase was formed as expected (Table). The proportion of the crystalline phase is higher due to the addition of Al_2O_3 in the KA mixture. The ratio of mullite to tridymite was the same for both mixtures (mullite/tridymite ~ 10.6). In the case of the analyzed kaolin, the authors did not find the presence of the andalusite and sillimanite phases.

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