In situ hyperspectral Raman imaging of the sintering process of kaolinite-bearing clay

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The phase transition of kaolinite to mullite is a well-studied process, because of its significance in the ceramic industry. The dehydroxylation reaction of kaolinite and the formation of mullite have previously been studied by thermal analyses and *ex situ* experiments [1]. *Ex situ* experiments, however, deliver mineralogical and textural information only after the sample has been cooled down to room temperature.

In the present study, hyperspectral Raman spectroscopic imaging has been utilized to *in situ* study the sintering process of kaolinite green bodies containing 35 vol.% of K-feldspar and 18 vol.% quartz, as determined by X-ray diffraction and Rietveld analysis. The clay was progressively fired at various temperature steps in air atmosphere in a Linkam heating stage from RT to 1300°C. Confocal micro-Raman spectra were recorded with a Horiba Scientific HR800 Raman spectrometer equipped with a 2 W Nd:YAG laser (532.09 nm) and an electron-multiplier CCD detector. A 600 grooves/mm grating was used, covering a wavenumber range from 100 to 1750 cm^{-1} in a single window. With this grating the spectral resolution was 3.5 cm^{-1}. Fast hyperspectral Raman images were recorded with a 1 μm pixel size using a 50 times LWD objective (N.A. = 0.8). The counting times varied between 0.1 to 0.5 s per pixel, which resulted in imaging times between about 5 to 25 min for a 50 x 50 μm-sized image.

Below 800°C the kaolinite breaks down to a dehydroxylated disordered metakaolin phase. Between 800 and 900°C K-feldpar, quartz, and the metakaolin react to form an Al-Si spinell phase that is stable up to the maximum temperature of 1300°C. The reaction could be followed at the grain scale. In addition, corundum and a silicate melt formed at temperature above 1000°C.

Our preliniary data are encouraging and suggest that i*n situ* hyperspectral Raman imaging is a useful method for the *in situ* research of the phase transitions and recrystallization processes at grain boundaries that take place during the thermal treatment of ceramic precursor materials.

[1] Gasparini *et al*. (2013) *Applied Clay Science* **80-81**, 417-425.