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The lizardite phase transformation followed by in situ high-temperature Raman and FTIR spectroscopy

R. Trittschack (1), B. Grobéty (1), and M. Koch-Müller (2)

(1) Department of Geosciences, University of Fribourg, Switzerland (roy.trittschack@unifr.ch), (2) Chemistry and Physics of Earth Materials, GFZ Potsdam, Germany

Inertialisation of chrysotile and CO_2sequestration in ultramafitesare examples of processes in which dehydroxylation of serpentine phases play an important role. Although research on the mechanism of this process goes back to the early 20th century, the dehydroxylation mechanism is still not fully elucidated. This study investigates the phase transformation of lizardite using *in situ* high-temperature micro-Raman spectroscopy as well as *in situ* micro-FTIR spectroscopy between room temperature and 819°C in order to demonstrate the temperature-dependent behaviour in the low-frequency region (100-1200 cm^{-1}) with bands assigned to Si-O and Mg-(O,OH) vibrations (Raman) as well as the high-frequency region (3400-3800 cm^{-1}) with hydroxyl stretching vibrations (FTIR and Raman). A thin film pressed in a diamond anvil cell was used for FTIR spectroscopy, whereas Raman spectroscopy was carried out on a loose lizardite powder. The spectra have been recorded at 25°C increments in the temperature range where dehydroxylation occurs, i.e. between 400 and 665°C. The *in situ* recording guarantees that the spectra are not affected by structural changes during quenching. All observed room temperature bands can be assigned to lizardite, although the OH-bands show a remarkable multiplication which vanishes with heating except of one extra OH band at 3665 cm^{-1}. The Raman active bands of lizardite show a linear shift to lower wavenumbers with increasing temperature. All Raman bands, except of a broad band at around 670 cm^{-1} diagnostic for Si-O-Si related vibration modes, disappear at a temperature between 639 and 665°C. Some of them exhibiting considerable frequency jumps in the last stage. We did not observe any water molecule related modes. Such band should be present assuming the conventional dehydroxylation model with water as only product (Zhang et al., 2010). The only band, which shows an increase in intensity during the dehydroxylation is the extra, non-assigned OH-band. Assuming that this increase is not a deconvolution artefact, the extra OH-band may be related to non-structural OH-groups that form during the transport of the dehydroxylation products to the grain surface. A hydrogen/oxygen/hydroxyl hopping mechanism has been proposed as alternative transport mechanism to water diffusion (Zhang et al., 2005). The frequency jumps and the appearance of a new band at 183 cm^{-1} are evidence for a hydroxyl bearing intermediate, partially ordered phase just before the formation of forsterite. The frequency of the new band is close to the diagnostic 189 cm^{-1} mode of the 10Å phase (Fumagalli et al., 2010). The formation of a possible dehydroxylation intermediate with a T-O-T arrangement was already proposed by MacKenzie and Meinhold (1994). Remaining Si-O-Si bonds are also pointing to the presence of a continuous T-sheet.

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