

Further complexities of the 10 Å phase revealed by infrared spectroscopy and X-ray diffraction

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ABSTRACT

Infrared spectroscopy and X-ray diffraction are used to evaluate the OH and H₂O environments in 10 Å phase (“TAP”), nominally Mg₃Si₄O₁₀(OH)₂·H₂O. Two partially deuterated samples of TAP synthesized under different conditions have very similar IR spectra, indicating that the phase has a reproducible structural state. IR spectra were also collected of samples of fully Ni-substituted and partially deuterated TAP, and of samples heated for 1–2 h at 500 °C to remove structural H₂O/D₂O and leave behind bands due to OH/OD of the 2:1 layer. A high-pressure study of the Ni-TAP sample confirmed that the behavior of its H₂O and OH/OD bands was analogous to that observed in previous studies of Mg-TAP. Comparison of the IR spectra of unheated, heated, and compressed samples has allowed three different types of Mg-OH (Mg-OD) stretching bands to be identified, two of which are further split, indicating subtle complexities in the TAP structure. The third band is identical to the band in talc. Two interlayer H₂O stretching bands have been identified. The presence of an absorption feature that is broader than these interlayer H₂O bands suggests that there is a second type of more weakly bonded H₂O. On heating to 500 °C, the main interlayer H₂O bands are lost, the talc-like band is unchanged, and shifts in the other Mg-OH band frequencies indicate a change in environment following the loss of the interlayer H₂O. At the same time the signature of a silanol group is possibly revealed from the coincidence of band positions in the Mg-TAP and Ni-TAP spectra. The recognition of three distinct Mg-OH (Ni-OH) environments in Mg-TAP (Ni-TAP) is consistent with the structural model of TAP proposed by Welch et al. (2006) and Phillips et al. (2007), in which the transformation from talc to TAP involves a key change from hydrophobic to hydrophilic character that enables hydration of the interlayer. A final level of complexity is indicated by the identification of a 3c trigonal superstructure from single-crystal XRD, implying a structure analogous to that of the 3T phengite polytype, with interlayer H₂O fulfilling the role of K. The formation of additional OH groups when talc transforms to 10 Å phase increases the amount of water contained in 10 Å phase and may also occur in closely related phyllosilicates in the Earth’s mantle, such as intergrowths of chlorite with 10 Å phase. Moreover, the reproducibility of the key features of the IR spectra for different samples implies that this water content is fixed.

Keywords: 10 Å phase, crystal structure, IR spectroscopy, XRD data, high-pressure studies, high-temperature studies