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Synthesis and characterization of D-bearing piemontite

Masahide Akasaka[1*], Mariko Nagashima[2], Maki Hamada[1], Asami Sano[3], Terumi Ejima[1]

[1]Shimane University, [2]Yamaguchi University, [3]Japan Atomic Energy Agency

Ca_[2]Al_[3-p]Mnˆ[3+]_[p]Si_[3]O_[12](OD)-piemontites were synthesized at 0.3-0.4 GPa and 500 C using hydrothermal equipment. The run products were analyzed using electron microprobe analyzer and infrared absorption spectral method, and refined using X-ray and neutron powder diffraction methods. The starting materials (s.m.s) of oxide mixture of stoichiometric compositions with p = 0.75, 1.0 and 1.1 and D_[2]O were used for hydrothermal synthesis experiments at 0.3-0.4 GPa and 500 deg.C. Starging materials: Oxide mixtures heated at 850 deg.C, and excess D_[2]O. Capsule systems: 1) Ag outer capsule with MnO_[2]-Mn_[2]O_[3] buffer + H_[2]O; Ag90Pd10 inner capsule with charge + D_[2]O; 2) Au outer capsule with MnO_[2]-Mn_[2]O_[3] buffer + D_[2]O; Ag90Pd10 inner capsule with charge + D_[2]O.

In the present synthesis experiments of deuterium-bearing piemontite using Ag-outer capsule with solid buffer + H_[2]O, deuterium was considerably or completely replaced by hydrogen. On the other hand, in the experiments using Au-outer capsule with solid buffer + D_[2]O, D-bearing piemontite was synthesized successfully. However, even in this case, seal of Au-outer capsule should be perfect, because hydrogen moves in and out inner Ag90Pd10 capsule.

There is no essential difference for Mnˆ[3+] distributions among octahedral M1, M2 and M3 sites between D-bearing piemontite and H-bearing piemontite: the Mnˆ[3+] occupancies at M1 and M3 in D-bearing piemontite refined using X-ray powder diffraction data are close to those of H-bearing piemontite (Nagashima and Akasaka, 2004). In this study, neutron diffraction data of a D-bearing piemontite synthesized from p = 1.0 starting material at 0.3 GPa and 500 deg.C were measured. Although D in this piemontite was considerably replaced by H because of the use of Ag-outer capsule with solid buffer + H_[2]O, D-positions could be refined. Neutron diffraction study of D-bearing piemontite synthesized successfully in this study will give us useful information for the relations between cation substitutions and hydrogen bond.

Keywords: piemontite, hydrothermal synthesis, infra-red, Rietveld analysis, deuterium