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Single crystal synthesis of δ-(Al,Fe)OOH

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Abstract

Single crystals of δ-AlOOH, δ-($Al_{0.953}$,$Fe_{0.047}$)OOH, and δ-($Al_{0.878}$,$Fe_{0.122}$)OOH with dimensions up to ~0.4–0.6 mm were synthesized by the high-pressure hydrothermal method. Synthesis experiments were performed at 21 GPa and 1470 K for 4 h using a Kawai-type multi-anvil apparatus. The crystals of δ-AlOOH, δ-($Al_{0.953}$,$Fe_{0.047}$)OOH, and δ-($Al_{0.878}$,$Fe_{0.122}$)OOH were colorless, yellowish green, and brown, respectively. Mössbauer spectra showed 95–100% Fe^{3+}/ΣFe at the octahedral site in δ-(Al,Fe)OOH. Chemical compositions of δ-($Al_{0.953}$,$Fe_{0.047}$)OOH and δ-($Al_{0.878}$,$Fe_{0.122}$)OOH are homogeneous with Fe/(Al+Fe) of 0.0469(8) and 0.122(3), respectively. Unit-cell parameters of δ-AlOOH are consistent with those of previous studies, and they increase with Fe/(Al+Fe). These results confirm that δ-AlOOH can form a solid solution with ε-FeOOH. The crystals contained a small number of fluid inclusions. The syntheses of large single crystals of δ-(Al,Fe)OOH will facilitate investigation of their phase stability, physical properties including elasticity and elastic anisotropy, behavior of hydrogen bonding, and spin state of Fe, which will improve models of the water and oxygen cycles in the deep Earth.

Keywords: δ-AlOOH, δ-(Al,Fe)OOH, water, single crystal, hydrous mineral, high-pressure synthesis, Kawai-type multi-anvil apparatus