Single crystal synthesis of δ-(Al,Fe)OOH using multi-anvil apparatus

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δ-AlOOH is an important hydrous mineral for understanding the water cycle in the deep Earth. In a descending slab, δ-AlOOH forms a solid solution with Phase H ($MgSiO_4H_2$) and ε-FeOOH [e.g., 1, 2]. This solid solution can transport water stored in its crystal structure to the deep mantle because it can be stable at the lowermost mantle conditions [1, 3]. Therefore, δ-AlOOH - Phase H - ε-FeOOH solid solution may affect the Earth's deep water cycles, chemical heterogeneity, and anomalies of seismic wave velocities at the lower mantle.
However, the stability, structure, elasticity, and spin state of this solid solution that are essential to discuss the issues above have not been constrained experimentally because of the difficulty to synthesize this solid solution as a homogeneous single phase.

In this study, we successfully synthesized Fe-bearing δ-AlOOH (δ-(Al, Fe)OOH) single crystals.
Single crystals of pure δ-AlOOH and δ-(Al, Fe)OOH with dimensions up to ˜0.6 mm were synthesized by a high-pressure hydrothermal method. Synthesis experiments were performed at 21 GPa and 1480 K for 4 h using a Kawai-type multianvil apparatus. Mössbauer spectra showed 95-100% $Fe^{3+}/\Sigma Fe$ at the octahedral site in δ-(Al, Fe)OOH. Unit-cell parameters of δ-AlOOH were consistent with those of previous studies, and they increased linearly with Fe/(Al+Fe) of the starting materials. The crystals contain 1-2 wt.% of excess water compared to their ideal water content. The syntheses of large single crystals of δ-(Al, Fe)OOH will facilitate investigations of their stability, elasticity, elastic anisotropy, spin state, and behavior of hydrogen bonding, which will improve our understanding of the water cycles, chemical heterogeneity, and anomalies of V_P and V_S in the deep Earth.

This work was supported by the JSPS Japanese-German Graduate Externship.

Reference
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Keywords: Water Cycle in the Earth's Interior, High Pressure and High Temperature Experiment, δ-AlOOH, ε-FeOOH, Single Crystal, Kawai-type Multianvil Apparatus

