

High-pressure behavior of ϵ -FeOOH inferred from experimental study

O. Ikeda^{1*}, T. Sakamaki¹, A. Suzuki¹

¹Department of Earth and Planetary Materials Science, Graduate School of Science,
Tohoku University, Sendai 980-8578, Japan

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*e-mail: osamu.ikeda.p5@dc.tohoku.ac.jp

Water covers about 70% of the Earth's surface and a large amount of them is thought to be carried into the inside of the Earth through slab subductions. Hydrous minerals (and/or phases) which are stable under high-pressure and high-temperature conditions can play roles as carriers of water, and hence have been investigated about their stability fields and physicochemical properties for understanding the behaviors of hydrous phases under high pressure. ϵ -FeOOH is one of rare phases to be stable up to even lower mantle condition where the pressure and temperature are exclusively high. ϵ -FeOOH is isostructural to δ -AlOOH, and a first principle calculation has indicated pressure-induced hydrogen bond symmetrization at 43 GPa and spin transition of iron at 65 GPa [1]. These transitions cause changes in physical properties; for example, symmetrization-related change of the longitudinal sound velocity and adiabatic bulk modulus of ϵ -FeOOH have been observed around 18 GPa [2]. Experimental studies of ϵ -FeOOH, however, are limited above 20 GPa and further measurements are needed for better understanding of pressure-induced changes.

We will report the results of two high-pressure experiments: 1) Raman spectroscopy and 2) inelastic X-ray scattering (IXS). The sample is polycrystalline ϵ -FeOOH, which was synthesized in advance from a powder of reagent grade 99 % α -FeOOH at the pressure-temperature condition of 8 GPa and 480 °C for two hours. For high-pressure generation, we used symmetric diamond anvil cells with 100 ~ 300 μ m culet sizes. The synthesized sample was loaded in the sample chambers of tungsten gasket.

For Raman spectroscopy, a spectrometer NRS-4100TOR (JASCO Corporation), which has a laser with the wavelength 532 nm and power 50 mW installed in Tohoku University, was used to obtain Raman spectra. The pressure in the chamber was calculated by means of the ruby luminescence pressure scale [3]. Along compression, the frequencies of most of Raman peak tops have positive pressure dependences but some peaks

(298, 386, 698, and 1099 cm^{-1}) changed the dependences to negative around 14 GPa. Above 15 GPa some Raman peaks (197, 298, and 443 cm^{-1}) disappeared and the others (364, 386, 549, 646, 698, and 1099 cm^{-1}) jumped by frequency 10–30 cm^{-1} . This change around 15 GPa may correspond to the replacement of vibrational modes in Raman spectra of δ -AlOOH at the pressure of 5.6 GPa [4], which indicates a hydrogen-bond related space-group transition from $P2_1nm$ to $Pnmm$ [5].

IXS spectroscopy was performed with X-ray diffraction (XRD) experiments at the BL35XU beamline of the SPring-8 facility in Japan. In this experiment, the sample was sandwiched between two foils of NaCl, which play as both of pressure medium and pressure marker. The XRD spectra of the sample and NaCl were obtained using a flat panel area detector installed in the beamline. We obtained the phonon dispersion of the sample at each experimental condition and calculated the velocity. Raman spectra in higher pressure range and sound velocities from IXS spectra will be discussed in detail at the meeting.

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