Phase boundary of FeOOH at high pressure and temperature

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1 Introduction

The sea covers 70% of the Earth's surface, and some of the abundant water is brought into the mantle by subducting slabs. Water affects the dynamics of the mantle, thus it is essential to investigate the physical properties of water carriers.

A high-pressure hydrous phase ε-FeOOH is a candidate of water carriers into the lower mantle, where the pressure and temperature are so high that most of other hydrous minerals decompose and release water. ε-FeOOH is a high-pressure phase of α-FeOOH goethite, and both of their crystal structures consist of the chains of FeO_6 octahedra (Fig. 1). The phase boundary between α and ε-FeOOH under pressure and temperature has been reported by several groups [1-4], but there is a large pressure inconsistency (~3 GPa) among them. In this study we have intended to determine the boundary precisely by high-pressure and high-temperature (HPHT) experiments.
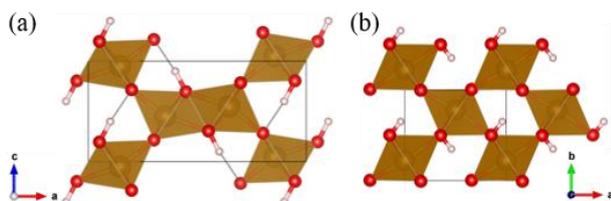

Fig. 1: Crystal structures of (a) α-FeOOH (b) ε-FeOOH. The chains of FeO_6 octahedra extend in the direction perpendicular to the sheet.

2 Experiment

The sample was a mixture of ε-FeOOH, regent grade NaCl and α-FeOOH. ε-FeOOH was synthesized in advance under HPHT at Tohoku University.

An X-ray diffraction (XRD) study under HPHT conditions was conducted at station AR-NE5C. The cubic type multi-anvil apparatus (MAX80) was used for HPHT generation. The high-pressure cell was an MA 6-6 type assembly with a pressure medium of boron and epoxy resin and a cylindrical graphite heater. An energy-dispersive method using a pure Ge solid-state detector was used to collect the XRD patterns.

The temperature was monitored with a W3%Re/ W25%Re thermocouple, and the pressure was determined using the equation of state of NaCl [5].

3 Results and Discussion

The stable phase at each HPHT condition was examined using time-resolved XRD in the pressure and temperature ranges of 4–7 GPa and 500–680 K, respectively. The sample was compressed to a desired pressure at room temperature, then was heated. Temperature was kept at every 25 or 50 K during heating up, and the durations of

retention were from several minutes to several hours depending on the reaction rate. We judged the stable phase at each HPHT condition from changes in the areas of the XRD peaks.

The XRD patterns obtained at pressure and temperature of 4.5 GPa and 625 K, respectively, are shown in Fig. 2 as an example. The peaks of α-FeOOH grew and those of ε-FeOOH declined, thus α-FeOOH is the stable phase at the condition.

We succeeded in observing both transitions from α to ε and from ε to α. Transitions were not observed at temperatures less than 575 K. The phase boundary is expressed as follows, assuming that it is liner.

$$P(GPa) = (-0.009 \pm 0.025) \times T(K) + (11 \pm 16)$$

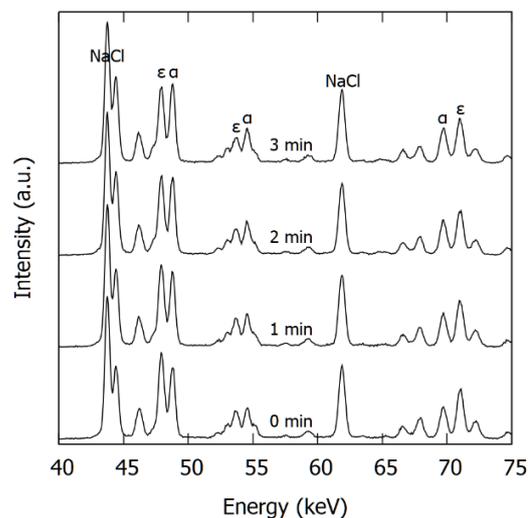Fig. 2: Time-resolved XRD patterns at pressure and temperature of 4.5 GPa and 625 K. α: the peaks of α-FeOOH; ε: the peaks of ε-FeOOH.

References

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