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In-situ X-ray observation of individual grains during grain growth of high-pressure ice VI

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Introduction

Rheology of planetary ices has an important key role on evolution, internal structure, internal convection, and tectonics of icy moons and planets of the outer solar system. Recent plastic deformation experiments on water ices have suggested that the grain-size sensitive creep is a dominant deformation mechanism under low stress conditions of planetary interiors. Therefore, it is important to constrain the grain size of ice that possibly controls viscosities of outer ice shell and icy mantle. The grain-size evolution in convective mantle depends on several processes such as dynamic recrystallization, grain-size reduction due to phase transitions, and grain growth. In this study, we observe the grain-size changes of high-pressure ice VI by in-situ X-ray diffraction.

Results and discussion

High-pressure phases of water ice are major constituents of the interiors of low-density icy moons with radius >700 km. Ice VI is stable at the base of the icy mantle (if differentiated) of large icy moons of Ganymede, Callisto, and Titan. Grain growth experiments of high-pressure ice VI were carried out by in-situ X-ray diffraction method using diamond anvil cell at BL13A and 18C, KEK-PF. We used monochromatic X-ray (29 keV, collimated to 100 microns in diameter) and obtained time-resolved 2D diffraction patterns every 10-20 minutes using imaging plate (Fig. 1). We have observed grain growth processes of polycrystalline ice VI from changes of the number of diffraction spots in the 2D diffraction patterns. Fine grained ice VI was synthesized at about 2 GPa and 250 K, and subsequently annealed at 300-350 K. The numbers of diffraction spots did not change 300 K for 20 hours, and slightly decreased at 320 K for 3 hours. At 350 K, the number of diffraction spots rapidly decreased and the intensity of each spot increased (Fig. 2). Based on optical microscopic observation, grain size of ice VI synthesized at 250 K was too fine to be observed, whereas after the annealing at 350 K for 75 min, the grain size of ice VI was increased to be about 60-70 microns. By counting the number of diffraction spots as a function of time, the evolution of the number of ice VI grains in reflection is obtained. The observed number of grains that fulfill the Bragg condition is proportional to the grain density, and the intensity of each spot is proportional to the volume of the grain. We preliminary estimated grain growth kinetics of ice VI from quantitative analysis of time-resolved 2D diffraction data obtained.

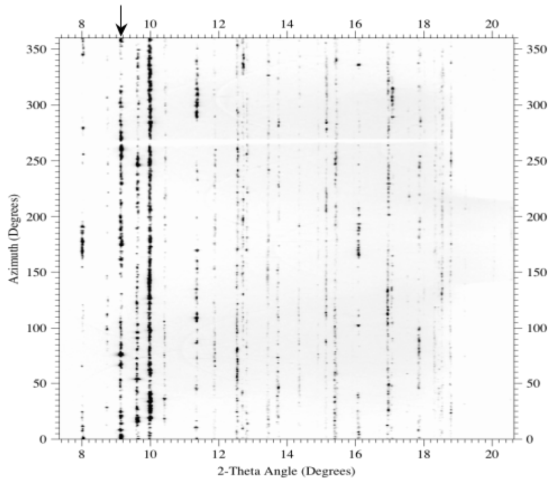

Fig. 1 2D diffraction pattern of polycrystalline ice VI obtained at 300K and 2.1 GPa by using DAC and imaging plate. Changes of (201) diffraction spot (arrow) density with time were preliminary analyzed as shown in Fig. 2.

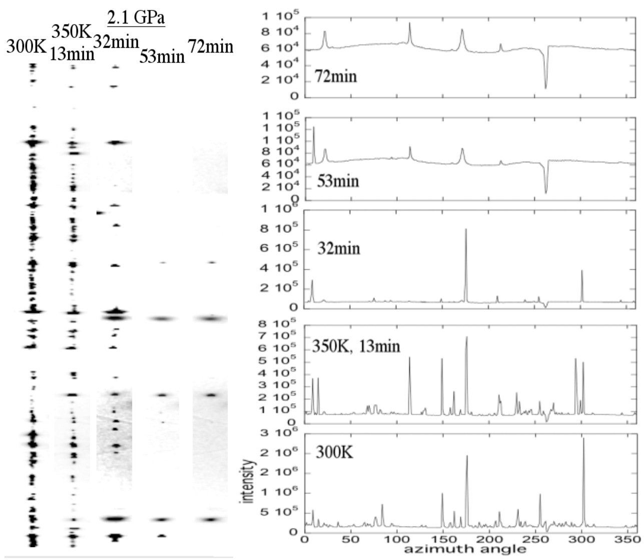

Fig. 2 Changes of (201) diffraction spot density with time during grain growth at 2.1 GPa and 350K (left). Integrated intensities of diffraction spot are shown with azimuth angle, in which each peak is identical to individual grain (right). The number and the intensity of peaks are proportional to the grain density and the grain volume, respectively.

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