Reinvestigation of site-disorder in dense ice by *in-situ* neutron diffraction techniques

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Ice VII is one of the crystalline ices that stably exist above 2 GPa at room temperature. Oxygens form a bcc-type lattice and each oxygen is bound to neighbouring oxygens via hydrogen bonds. Hydrogens are disordered among the four sites on the oxygen-centred tetrahedra with equivalent probability resulting in their occupancy of 0.5 shown in Fig. 1. This simple cubic structure model is widely adopted but the *true* structure of ice VII is yet to be known. Strictly speaking, the oxygen sublattice is not bcc, and two models with oxygen displacements along <100> [1] and along <111> [2] are postulated. We reinvestigated the site disorder of oxygens (and hydrogens) in ice VII by neutron diffraction using modern high-pressure apparatuses.

Single-crystal and powder neutron diffraction patterns were collected at the D9 at the ILL in France and at the BL11 (PLANET) at the MLF J-PARC in Japan, respectively. Both measurements were conducted at approximately 298 K and 2 GPa. The single-crystalline specimen were directly crystallised from an alcohol-water mixture (D_2O:MeOD:EtOD = 5:4:1 in vol. ratio) in a newly-developed diamond anvil cell [3]. Powder specimens were prepared from pure D_2O in situ using the MITO system [4]. Fine powder crystals were obtained through solid-solid phase transitions (ice I_h+III→II→VI→VIII→VII).

Single crystals of ice VII were obtained by cyclic heating and cooling at a pressure above 2 GPa. The collected diffraction patterns were analysed by the maximum entropy method. The obtained scattering length density map exhibited anisotropic distribution from the average site. A derived pair-distribution function resembles that calculated from the average structure model in the long-*r* region while it does not match in the short-*r* region. This inconsistency is considered to be caused by the correlation between local structures.

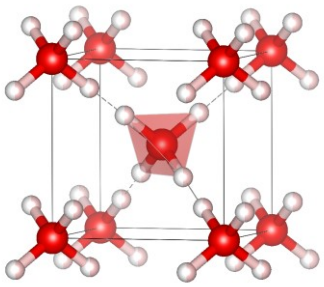

Figure 1. Average structure model of ice VII with cubic unit cell. Hydrogen sites (white) are 50% occupied.

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