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氷 VII 相のイオン抵抗及び屈折率の圧力応答
Pressure response of ionic resistance and refractive indices of ice VII

岡田 卓 [1*], 八木 健彦 [1], ミハイル・エルメッツ [2], イワン・トロヤン [2]
Taku Okada[1*], Takehiko Yagi[1], M.I. Eremets[2], I.A. Troyan[2]

[1] 東京大学物性研究所, [2] マックスプランク化学研究所
[1]ISSP, Univ. of Tokyo, [2]Max-Planck-Institut fur Chemie

Introduction

Ice VII is the stable form of ice at ambient temperature between 2.2 and ˜40 GPa. Loubeyre et al. (1999) has detected spatially modulated phases between 2.2 and 25 GPa by single-crystal X-ray diffraction. However, the inducement mechanism remains unknown. Cavazzoni et al (1999) has theoretically predicted a superionic phase of H2O with fast hydrogen diffusion between solid phase and fluid phase above 20 GPa and 2000 K. In this study, we attempted to measure ionic conductivities and refractive indices of H2O directly at high pressures and temperatures by using diamond anvil cells (DAC).

Experimental methods

We performed the experiments using a DAC with type I diamonds having flats of 0.35 mm in diameter. A rhenium gasket combined with cubic boron nitride powder as insulation material was used to contain the sample. Distilled and deionized H2O was loaded into ˜0.1 mm diameter and ˜0.03 mm thick sample chamber in the DAC. The pressure at room temperature was determined by the Raman spectrum of diamond-anvil. The central area of the sample of about 0.05 mm in diamter was subsequently heated from one side with a laser. The size of the heating spot was larger than the distance between the electrodes. Temperature was measured by a spectroradiometric method above 900 K and interpolated below 900 K from the relationship between the laser power and the temperature, respectively. We measured ionic resistance, namely, ac electrical resistance with an LCR meter (Agilent 4284A) and the platinum electrodes in configuration of a quasi four-probe microcircuit at high pressure and temperature. In our impedance spectroscopy (IS) measurement, an 1 V ac voltage signal was introduced into the system and the response of the system to this signal is expressed by complex impedance $Z = R + Xi$, where R and X are the real part and imaginary part of Z, respectively. The frequency ranged from 20 Hz to 1 MHz. During IS measurement, we kept the sample temperature by controlling the laser power. Optical path length of the sample was also measured at high pressure and ambient temperature.

Results and discussion

We observed ideal impedance arcs in comparative high frequency region. We fitted the R-X plot with a function of a semicircle and obtained a bulk resistance of the sample at different pressures and temperatures. At 2.2 GPa, the bulk resistance of ice VII was greater by the one order of magnitude of that of Ice VI. At ice VII stable region, the bulk resistance decreased with increasing pressure. At 10 GPa, it was minimum value and smaller by the one order of magnitude of that of ice VII at 2.2 GPa. Then, it increased with increasing pressure. At 20 GPa, it was almost identical with that at 2.2 GPa. Above 20 GPa, it decreased slightly with increasing pressure. The change with pressure at ambient temperature is very reproducible between the all five experimental runs. The pressure region of anomaly of the bulk resistance of ice VII is consistent with that of the modulated phases reported by Loubeyre et al. (1999). At around 45 GPa, the bulk resistance decreases gradually with increasing temperature and some discontinuous changes were observed. High temperature experiments to check the reproducibility of the observed results and to clarify the behavior at different pressures are now in progress.