K134-P002 **Room: Poster Session Hall** **Time: May 16**

In-situ observation and Raman spectroscopy of ice 4 under high pressure conditions

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1. Introduction

It is needless to say that water is very important for us. Since water is quite familiar to us, some people may think that all about water is already clarified. But in fact, we still have many questions about water. And ice, which is the easiest phase to understand the structure, is still widely studied to understand water.

It is known that ice shows more than 17 kinds of structure. The structure of the ice we always see is called ice 1h, and it is merely one of those many structures. In addition, there are two metastable ices. One of them is ice 4 (Bridgman, 1935). It is difficult to make due to its metastability, so we do not know much about it. And its state and Raman spectrum under high pressure is almost unknown (Chou and Haselton, Rev. High Pressure Sci. Tech., 1998). So, we tried to observe it and to measure the Raman spectrum under high pressure.

2. About Ice 4

As mentioned above, ice 4 is metastable ice. Differently from other ices, ice 4 has not its own stability field, and may exist where the temperature is lower than its melting curve. According to Chou and Haselton (1998), ice 4 is formed together with ice 1h at around 223K, starts melting at ice 4-ice 1h-liquid triple point, and finishes melting on its melting curve.

3. Experimental Procedures

We compressed water with diamond-anvil cell (DAC) and cooled it with liquid nitrogen. DAC is a high-pressure equipment which sandwiches a sample with two diamond-anvil faces and compress it. We measured pressure with ruby fluorescence, temperature with thermocouples, and Raman spectrum with micro Raman spectroscopy. Raman spectroscopy allows us to know structural information of substance through spectrum of scattered light.

4. Experimental Results

4-1. Raman Spectra

On determining Raman spectra of ice 4, the biggest difficulty was that they resemble to those of ice 6. The highest peak of Raman spectrum of ice 6 we measured is 3227cm^{-1} and that of ice 4 was 3206 cm^{-1}. The peak position is also a function of temperature and pressure. So we once thought that we failed, and nearly surrendered. But as we checked data we had obtained, we found that the Raman spectrum of an ice located in the stability field of ice 2 does not resemble to known spectrum of ice 2 (Chou et al., Science, 1998). And the spectrum is quite similar to that of the ice we thought as ice 4. Ice 4 can exist in the area of ice 2, and ice 6 cannot. Therefore we conclude that we obtain ice 4.

4-2. Melting Point

Since melting points of ice must be on the melting curve, data set of melting points is an important clue to identify a phase of ice. First, we plotted the melting point of ice 6 we measured on the phase diagram, and confirmed that it was on the melting curve of ice 6. Then we plotted the melting points of all ices we thought as ice 4 on the diagram, and all of them were on the melting curve of ice 4. (Chou and Haselton, 1998) And all spectra were similar to each other.

5. Conclusion

A Raman spectrum which is probably of ice 4 or ice 6 is obtained in the area of ice 2, and this spectrum is obviously different from that of ice 2. And all ices with the same spectra melted around the known melting curve of ice 4. Therefore we think we succeeded in making ice 4 and measuring its Raman spectrum. In this paper we report our experimental procedures and results shortly. In our presentation we will show Raman spectra, photos and movies. If you are interested in our experiments, you will be most welcome.

Acknowledgement

This study was initiated in a course of basic experiments for earth science at Kyoto University during fall semester of 2008 fiscal year with mainly freshmen including Mr. Masahiro Hayashi, Mr. Naomi Takakuwa, and Mr. Hideki Ikeda.

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