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Developments of pressure and temperature controlling system for x-ray and neutron scattering experiments

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Some polymorphs or amorphous phase of ice, for example, ice II, HDA, LDA, etc,, could only be accessible by compressing under low-temperature, and metastable ice phases like ice Ic, IV, XII, VII' are formed through HDA. Therefore, it is essentially important for the study of ices to control pressure and temperature individually. For x-ray study, diamond anvil cells (DACs) with helium-gas driven membrane have been widely used in synchrotron facilities so far, and the DAC often attaches to a helium compressor-type cryogenic system. The problems of cryostat are 1) generally too large and heavy, and 2) having noise and oscillation so that it is difficult to set it to laboratory based x-ray diffractometer. We have developed liquid-nitrogen circulating type cryogenic system with newly designed DAC with very large opening angle. The considerably small cryostat and the specially designed DAC allow us to conduct single crystal or powder x-ray diffraction experiments under pressure from 80 K to 473 K, and of course, pressure and temperature are individually controllable. Moreover, there is no oscillation thanks to removing pulsate compressor from the system.

On the other hand, for neutron experiments, high pressure (up to 2000 bar) helium-gas driven Paris-Edinburgh cells have been used in ISIS and ILL. Although this technic has been quite sophisticated at the moment, some technical difficulties like helium gas leaking and safety problems have still remained. Therefore, we again adopt liquid nitrogen circulating system for 100 ton opposed-type press for high pressure and low/high temperature neutron scattering experiments.

The p-T controlling system for x-ray has been now nearly perfected and that for neutron is going to be completed until March/2012. We reported the details of the systems and briefly introduce some preliminary studies using them.

Keywords: Low temperature, High pressure, ice, x-ray diffraction, neutron diffraction