

Hydrogen in portlandite -Neutron diffraction measurements at high pressure and high temperature-

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Ca(OH)₂ is one of the simplest hydrous minerals. Because this type of structure is a component unit in complex hydrous phases such as chondrodite, it is important to understand structural behaviors at high pressure and high temperature. However, only a few previous researches can be found on crystallography of Ca(OH)₂ in the conditions of simultaneously high pressure and high temperature, although there were quite a few studies on EOS, phase relation, melting and so on, which were performed by using X-ray diffraction technique. Last year a brand-new TOF neutron beamline (PLANET) dedicated for high pressure and high temperature measurements started to operate in J-PARC, Japan. Ca(OH)₂ was selected as one of the first targeted materials measured at the PLANET.

Deuterated samples were prepared via hydrothermal treatment with CaO fine powders and excess D₂O water in a Teflon lined stainless steel autoclave at 493 K for 4 days. After the hydrothermal treatment was completed, precipitates were filtered out, washed with D₂O water, and then dried at 383 K under vacuum for 3 hours. The products were confirmed to have the CdI₂-type structure by conventional powder X-ray diffraction measurements and were checked to be deuterated by IR absorption spectra. TOF neutron powder diffraction measurements of Ca(OH)₂ were carried out from 300 to 773 K at about 3 GPa at the PLANET beamline in J-PARC, Japan. At first, temperature was increased to 773 K at about 3 GPa for annealing and then data acquisition was carried out at each temperature condition to lower temperature. The measurement time at each targeted P-T conditions was about 8 hour after the temperature reached equilibrium. Generation of high pressure and high temperature can be performed by using the 6-ram big press (Atsuhime) installed at PLANET.

Quality of diffraction patterns is surprisingly superior and only diffraction peaks from Ca(OH)₂ could be observed owing to radial collimators equipped with Atsuhime, although powder sample of Ca(OH)₂ was loaded into a cylindrical graphite furnace in ZrO₂ pressure medium cube. The detailed structure parameters such as lattice parameters and atomic coordinates were refined by the Rietveld method by using a program GSAS.

Keywords: hydrogen, portlandite, high pressure and high temperature, neutron diffraction, Atsuhime, PLANET