Single crystal X-ray structure analysis of high pressure phase of Kalicinite

Kazuki Komatsu[1]; Hiroyuki Kagi[2]; Takaya Nagai[3]; Takahiro Kuribayashi[4]; John B. Parise[5]; Yasuhiro Kudoh[1]

[1] Tohoku Univ; [2] Lab. Earthquake Chem., Grad. School Sci. Univ. Tokyo; [3] Earth and Planetary Sciences, Hokkaido Univ.; [4] Tohoku Univ.; [5] State Univ. of New York

Kalicinite (KHCO3) single crystal which was grown from an aqueous solution by slow evaporating was compressed by diamond anvil high pressure cell, and the obtained high pressure phases were investigated by X-ray diffraction analysis. A modified Merrille-Bassett type diamond anvil cell was used for the high-pressure experiment. The SUS301 stainless steel plate in the center was used as a gasket. A 4:1 methanol-ethanol mixture was used for the pressure medium. Ruby chip for determining pressure was not used to exclude the extra spot. Pressure was approximately estimated by Raman band of methanol-ethanol mixture in the diamond anvil cell instead of Ruby fluorescence method. The C-C stretching mode of methanol-ethanol mixture was observed at 899 cm-1. Raman spectra of 4:1 methanol-ethanol mixture with increasing pressure were measured by Wang et al. (2004). Their results showed that the C-C stretching mode was observed at 882 cm-1 at 0 GPa, and linealy increased with increasing pressure with the pressure dependence of the band of 3.7 cm-1/GPa. Hence, pressure was estimated about 4.6 GPa. All X-ray diffraction measurement in this study was carried out at this pressure. Above the phase transition pressure, X-ray oscillation photographs were collected by an imaging plate X-ray diffractometer (Rigaku, R-AXIS IV++) with rotating anode (MoKa, 50kV, 80mA). The observed reflections were assigned by a monoclinic and a triclinic phase. These monoclinic and triclinic lattices were independent of ones of existing three polymorphs (phase I-III). Hereafter, the monoclinic phase would be called phase IV and the triclinic phase would be called phase V. X-ray diffraction measurement for determining the unit cell parameters and for collecting intensities for phase IV was performed using a four-circle X-ray diffractometer at the beam line BL-10A of Photon Factory, KEK, Japan. The structure was solved by direct methods. The non-hydrogen atoms were refined isotropically. The final R factor after full-matrix least-squares refinement on F was 10.0%. Two CO3 molecules in phase IV are order in an antiphase configuration the same as the ambient phases (phase I, II).