Crystal structure of high pressure Al(OH)3 polymorphs: I. Single crystal X-ray diffraction study of eta-Al(OH)3

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Minerals of the alumina-water system, which have the composition of Al(OH)3, are gibbsite[gamma-Al(OH)3], bayerite[alpha-Al(OH)3], nordstrandite[beta-Al(OH)3] and doyleite[no alphabetical notation]. Dachille and Gigl (1983) studied the system Al2O3-H2O using high-pressure opposed anvils apparatus in the temperature and pressure ranges 20-450 C and 0-6 GPa and found two new polymorphs as quenched phase, and they called it as delta-Al(OH)3 and beta'-Al(OH)3. According to Dachille and Gigl (1983), beta'-Al(OH)3 phase was only occurred using gibbsite as the starting material, and not occurred using other phase in the Al2O3-H2O system such as bayerite. In the past decade, the high pressure phase of gibbsite was investigated by powder X-ray diffraction (Huang et al., 1996, 1999; Liu et al., 2004) and Raman spectroscopy (Johnston et al., 2002). However, the crystal structure of this high pressure phase has been unknown until now. By the way, Dachille and Gigl (1983) recognized gibbsite as alpha-Al(OH)3, bayerite as beta-Al(OH)3 and nordstrandite as gamma-Al(OH)3. However, the standard alphabetical notation for Al(OH)3 polymorphs is ones mentioned at the beginning. Therefore, the name of beta'-Al(OH)3 seems not any more appropriate. We proposed that a new alphabetical notation, eta-Al(OH)3, is given for the high pressure phase of gibbsite instead of beta'-Al(OH)3. In this study, the crystal structure of eta-Al(OH)3, which was solved by single crystal X-ray diffraction method, was reported.

Single crystal of natural gibbsite from Langesundfjord, Oslo Region, Norway was loaded into a DAC with a fluorinert as a fluid pressure transmitting medium. No ruby chip for determining pressure was mounted to avoid the extra spots, but pressure and the phase transition could be detected by Raman spectra. Above, the phase transition pressure, X-ray oscillation photographs were taken using an imaging plate X-ray diffractometer (R-axis IV++, Rigaku) with rotating anode (MoKa, 50kV, 80mA). The obtained reflections of eta-Al(OH)3 could be indexed as a single phase, and hence, the phase transition from gibbsite to eta-Al(OH)3 is single crystal-single crystal phase transition. X-ray diffraction measurements for determining lattice constants and for collecting intensities of eta-Al(OH)3 was performed using a four-circle X-ray diffractometer installed at the beam line BL-10A, Photon Factory, KEK, Japan. The model of crystal structure except for H atoms could be solved by a direct method using the Sir2004 program package, and the structure refined using SHELXL. Accordingly, the R-factor is to be 6.25 %. The crystal structure of eta-Al(OH)3 consists of Al octahedral layer as well as other Al(OH)3 polymorphs except for delta-Al(OH)3.