In situ structural refinement of CaSiO3 perovskite under lower mantle condition

Takeyuki Uchida[1]; Yanbin Wang[1]; Norimasa Nishiyama[2]; Mark L. Rivers[1]; Steve R. Sutton[1]; Ken-ichi Funakoshi[3]; Akifumi Nozawa[3]; Tetsuo Irifune[4]; Toru Inoue[4]; Akihiro Yamada[4]; takehiro kunimoto[5]

[1] GSECARS, Univ. of Chicago; [2] Dept. Earth Sci., Ehime Univ.; [3] JASRI; [4] GRC, Ehime Univ.; [5] GRC,Ehime Univ.

CaSiO3 perovskite (Ca-Pv) is one of the most abundant components of the lower mantle (6-12 % by volume). High pressure and high temperature equation of state of Ca-Pv, therefore, has been studied extensively to discuss the structure and dynamics of the lower mantle. Density comparison of (Mg,Fe)SiO3 orthorhombic perovskite and Ca-Pv (in cubic symmetry; Pm3m) leads to the conclusion that Ca-Pv can be regarded as 'invisible' component in the lower mantle because the difference in density is too small to distinguish these two phases from seismography. In terms of symmetry of Ca-Pv, tetragonal or even lower symmetry has been proposed theoretically and experimentally using laser heated diamond anvil cell. The structure change in Ca-Pv may affect its shear modulus, suggesting that Ca-Pv could seismically play an important role, and thus examination of symmetry is of fundamental importance. We have been developing a new step-scan diffraction technique, which collects angle-dispersive data using energy-dispersive setup, solid-state detector (SSD) and collimator assembly with white X-rays. Our feasibility test of this technique has successfully been commissioned. We apply this technique to Ca-Pv sample under lower mantle conditions. High pressure and high temperature experiments are conducted on the bending magnet beamline (BL04B1) at SPring-8, using the 1500 ton multi-anvil press. The high pressure apparatus consists of eight cube-shaped inner anvils (14 mm edge length), made of polycrystalline diamond sintered by SiC or Co with truncated edge length of 2.0 mm, and of six outer 'DIA' type anvils that form a cubic cavity with an edge length of 27 mm. The goniometer consists of two arched rails in horizontal direction and rests on x-y-z translation stages, so the rotation centers of 2theta angle can be aligned to both horizontal and vertical center of the sample. Energy-dispersive signals are processed by multi-channel analyzer, which is calibrated using radioactive sources. The number of channels is set to 4096 and shaping time to 4 ms. The sample and the pressure calibrant (mixture of Au and MgO) are packed in separate layers in the sample chamber (1 mm diameter), at the center of pressure medium made of amorphous boron and epoxy resin. The cylindrical axis of the sample is located perpendicular to incident X-rays so goniometer scans in the radial direction of the sample, reducing a shape effect of the sample on diffraction intensity. A WRe thermocouple monitors temperature while internal resistance heaters made of TiC and diamond powder heats the sample and the calibrant. The entire EDD dataset is combined to form a two-dimensional array of intensities, each value corresponds to a given X (photon energy) and Y (two-theta) index. This 2-D dataset can be viewed according to a fixed X or Y value, corresponding to ADD (angle-dispersive diffraction) or EDD (energy-dispersive diffraction) spectra, respectively. By choosing the intensities at various 2theta values for certain fixed energies (wavelengths), a series of ADD patterns are obtained. To compensate coarse step size (0.05degrees) of the scans, several ADD are binned. For example, the ADD from 69.46 keV to 70.42 keV, corresponding to 21 channels, are binned to obtain 70.03 keV spectrum. These data are fit using the Rietveld refinement software package GSAS. Based on the refinement results, the 2-D diffraction data can also be reproduced from lattice parameters and extinction rules. Our results reveal that Ca-Pv has extra Bragg's peaks under lower mantle condition, supporting lower symmetry than Pm3m. Space groups Cmcm, I4/mcm, I4/mmm, Imma, P4/mbm, P4/mmm, P4/nbm, and Pbnm are being examined to conclude the possible structure change in Ca-Pv.