Structure analysis of a $SrTiO_3$ perovskite single crystal at 3.5GPa using hard x-rays of 30 keV

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$SrTiO_3$ is an ideal cubic perovskite with the lattice constant of a = 3.908 Å. Ti and Sr atoms occupy the octahedral site (B site) and 12-fold coordination site (A site), respectively. Structure analysis of the crystals including transition metal elements is important to comprehend the electronic state especially under high pressure. Many reflections are required in order to clarify the electron density by an x-ray diffraction study. However, generally a diamond anvil cell (DAC) has a limitation in the measurable reciprocal space and the number of reflections because of its geometry. Therefore, high energy x-ray source providing short wave lengths is desirable for the single crystal diffraction study using a DAC. The beamline facility of a seven-circle diffractometer at BL02B1 designed for the single crystal x-ray diffraction experiments in the energy range from 5 to 50 keV has a great advantage for this purpose.

This study was performed using hard x-ray radiation of 30 keV, which was monochromatized by silicon 111 double crystals and focused by mirrors. The energies were calibrated by the Sn K-edge (29.192keV). A synthetic single crystal of $SrTiO_3$ perovskite with the size of $80\times80\times80$ μm^3 was compressed together with the 4:1 mixture of methanol:ethanol pressure medium. The pressure was determined by a conventional ruby R1 fluorescence method and was estimated as 3.5 GPa. X-ray diffraction intensities were measured by a scintillation counter which is installed in a distance of 500 mm from the goniometer center.

Firstly, we took an imaging plate (IP) photograph with a vacuum camera in order to search reflections of the crystal. The 2theta and the omega axes were fixed and the phi axis oscillated within $\pm20°$. The phi rotation speed was 8° /min and the exposure was repeated twice. From the diffraction spots on the IP, we calculated the values of 2 theta, omega, chi, and phi. Peak-search and peak-refine operation were executed by scanning the four axes. At first, an UB matrix was estimated by vector minimum method using five observed reflections. Subsequently, 16 reflections were used to determine the final UB matrix by least-squares method. The size of an incident pin-hole slit and the opening angle of a receiving slit for these peaks refining were 500 $\mu m\phi$ and 1°, respectively.

The refined cell parameters of $SrTiO_3$ at 3.5 GPa were shown in Table 1. The parameters were not constrained as a cubic system in order to check the accuracy of cell refinements. The parameters listed in Table 1 have very large estimated standard deviation and don't indicate a cubic lattice constant which we have expected to be. This is because some peak positions were not determined accurately due to the unexpected poor crystallinity of the sample. And centering of peak positions was made not by a computer program automatically but by hand. Moreover, the setting of PHA and HV of the scintillation counter might not be good enough. Indeed, counts per second of a same index reflection which was measured at BL02B1 were nearly equal to a data which was measured at our laboratory (using Mo rotating anode x-ray generator, 45kV, 150mA).

Table 1 Cell parameters at 3.5 GPa.

a/Å	3.8684(95)
b/Å	3.8762(45)
c/Å	3.8972(108)
α/°	90.345(278)
β/°	89.933(255)
γ/°	90.106(173)

Acknowledgment: We are grateful to members of Prof. Noda's group, Chiba University for their technical assistance in the experiments.