First Experiments at BL02B1 and Some Results on Structural Phase Transition SG

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1. First Experiments

First experiments, as the start-up experiment at BL02B1, were performed during five days just before the summer shut-down in 1997. The measurements were carried out under the collaboration of 4 subgroups participating to this beam line. The following reports were obtained in these experiments, and they were given at SRI'97 and will be published in ref.[1].

· *XAFS measurements*
· *Si single and powder diffraction*
· *vacuum camera*

The absorption coefficient of Ag is shown in Fig.1 as a function of energy. The XAFS pattern seems good, even around fairly high energies, such as 25keV and 30keV. We evaluated the accuracy of the monochromatized energy. The monochromator gives an accurate energy, within 30 to 50eV. At the present time, the actual energy is slightly higher than that requested.

A diffraction pattern from a Si single crystal was taken to test the accuracy and the reproducibility of the diffractometer. Reflections from 220 to 10 10 0 were taken with the 0.0001 degree minimum-angle-increment of the w-axis. The data were taken when the ring current was 1mA, and we used a 1/20 attenuator to reduce the intensity. The observed width was 0.002 degree FWHM, and the obtained diffraction pattern was well re produced with the accuracy within 0.0002 degree. We calculated the wavelength of the incident X-rays, by using the known lattice constant of the silicon sample. The result was 0.49948(1) Å for a requested setting of 0.50000Å. This value is consistent with the result of the XAFS measurements.

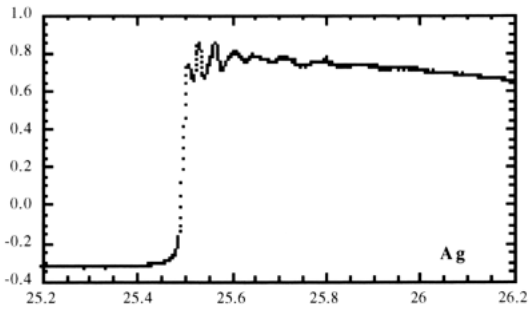

Fig.1 XAFS measurements at the energy around the absorption in Ag.

We took several Si powder reflections, up to 13 7 1 to test the soller slit, when the ring current was 18mA. In Fig. 2, powder diffraction profiles of Si are shown, in which the incident wave length is settled to be 0.50000Å. The widths observed are 0.04 degree for all peaks. The consistency of the observed peak positions with calculated ones was satisfactory and the differences were within 0.005 degree.

We tested the vacuum camera with several crystals to evaluate its capabilities. We took a photograph using a metal-organic crystal $[Ni(C_6H_{14}N_2)_2][PtBr_2(C_6H_{14}N_2)_2]Br_4$ which is characterized by a Pt-Ni chain structure. The crystal size was 0.3x0.1x0.05 mm^3, the rotation angle was 10 degrees and the exposure time was 20 minutes. The diffraction spots were clearly seen, and the dif-fuse lines

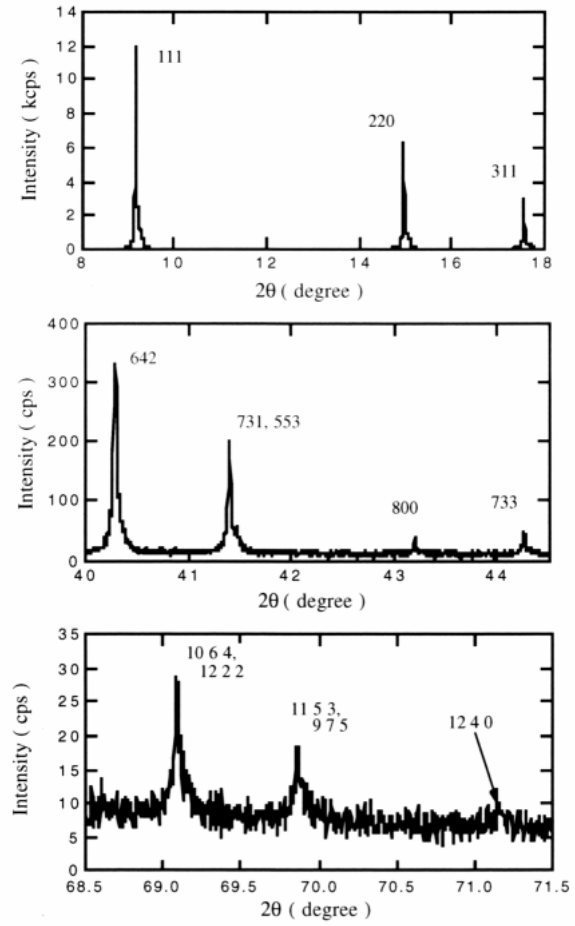

Fig.2 Si powder diffraction profiles.

were also seen, which originated from the disordered character of the Pt-Ni chains. We consider that a structure analysis with a 10mm crystal is possible if the ring current is increased to 100mA and focusing of the beam by the monochromator and mirrors is realized. We used a standard type monochromator of SPring-8 with a Si crystal. The beam height is 1400mm before the monochromator and 1430mm after the monochromator. Two mirrors will be settled-in at the beginning of 1998, to eliminate the higher order harmonics and to focus the beam for lower energy range (5<E<22keV). When the mirrors are used, the beam height changes from 1525mm(22keV) to 1640mm (5keV).

2. The Activity of Structural Phase Transition SG after October 1997

We studied the phase transition of hexagonal $BaTiO_3$, partially in order to tune the counter system with the energy from 5keV to 70keV and to test a cryostat. High resolution powder diffraction experiments were carried out to determine the symmetry of phase III of h-BT. First, we took a Weissenberg photograph with a single crystal at the laboratory X-ray system. In phase II and phase III, we found no superlattice reflections so that we consider that the relevant modes for the phase transitions are the G-point modes and there is no cell doubling for both phase transitions. The unit cell and the space group of phase II were confirmed to be the orthorhombic and $C222_1$. The symmetry in phase III is newly determined as a monoclinic with the deformation of the g-angle(about 0.05˚) by the synchrotron radiation. The most possible symmetry in phase III is $C112_1$, or equivalently $P112_1$ as a crystallographic conventional unit cell. In Fig.3, we show the powder diffraction as a function of temperature. At room temperature, the best resolution as a FWHM is D2q=0.0085˚ at 2q=24˚, since we used an analyzer crystal instead of a soller slit. Preliminary results will be published in ref. [2].

We also took an oscillation photograph of $La_{2-x}Sr_xCuO_4$ with high energy X-rays, such as 35keV and 70keV, as shown in Fig.4. Theses energies were chosen so that the absorption coefficients become suitable. We could obtain good photographs as shown in Fig. 4. The high energy experiments is definitely a powerful tool at SPring-8.

References

[1] Y. Noda, K. Oshima,H.Toraya, K.Tanaka, H. Terauchi, H. Maeta and H.Konishi: J. Synchrotron Radiation 5(1998) in press.

[2] Y. Noda, K. Ayama,T.Shobu,Y.Kuroiwa, H. Nakao, Y. Morii and H.Yamaguchi: to be publish in Ferroelectrics (1998).

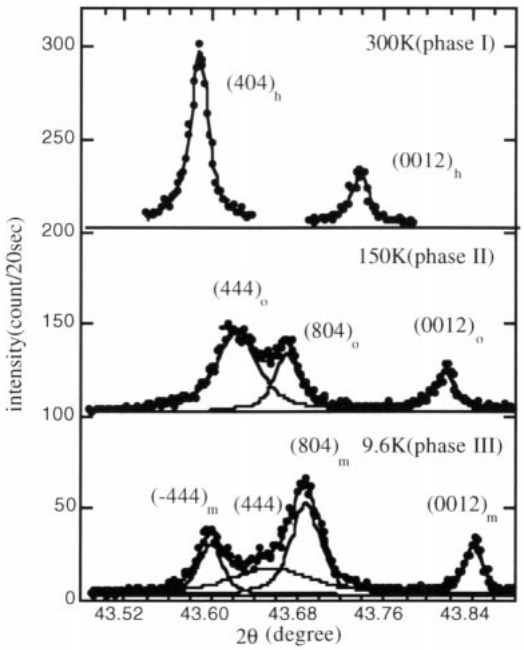

Fig.3 Powder diffraction of h-BT in Phase I, II and III.

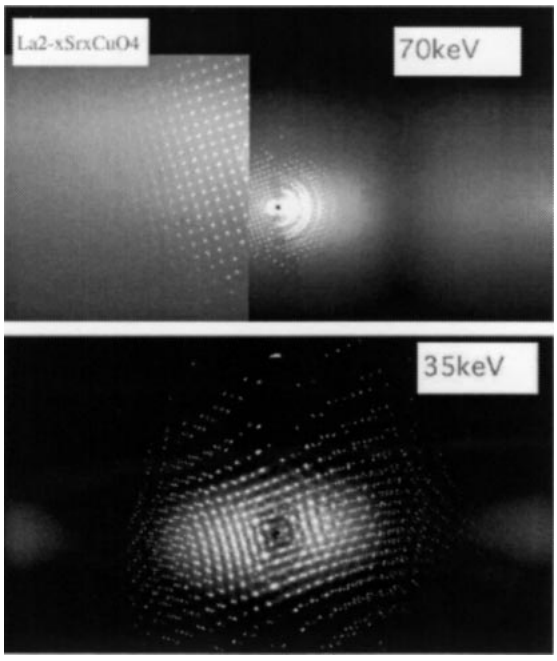

Fig.4 Oscillation photograph of La2-xSrxCuO4 taken with a vacuum camera.