Study on the phase transition of CTFP using Wide-band X-ray beam

T. Koganezawa, T. Udagawa, Y.Yoshimura, N.Nakamura and H.Iwasaki,

1. Introduction

A new diffraction system was built at the Synchrotron Radiation Center at Ritsumeikan University (BL-1: beamline 1), in which a parallel X-ray beam of a band of wavelengths is produced by reflection from a multilayer monochromator of depth-graded thicknesses last year [1]. The bandwidth is 600eV as shown in Fig.1, useful photon energy range is from 6000eV to 8000eV. It provided with a sample cooling/heating system and is useful to study an X-ray diffraction experiments in which energy-dispersive intensity profile of Bragg reflection is measured. The beamline is designed not only for the wavelength modulated diffraction (WMD)[2] study but also for ordinary structural studies of a crystal by X-ray diffraction experiments. This short note reports on a new application to a study of a phase transition using a stationary crystal diffraction method keeping the wide-band parallel X-ray beam.

2. Experimental and results

Cholesteryl 2,2,3,3-tetrafluoropropionate (CTEP: $C_{27}H_{45}OCOF_2CF_2H$) crystal undergoes successive phase transitions at 178, 143 and 123 K [3]. Crystal data are monoclinic with space group $P2_1$ or $P2_1/m$, and lattice constants a=1.244, b=0.927, c=1.330 nm and β=106.0˚ at room temperature.

The diffractometer with the sample cooling/heating system of the beamline is equipped with a two-dimensional detector of a curved imaging plate, and is installed to carry out diffraction experiments in the fields of structure determination for a single crystal and of a phase transition induced by changing temperature. A series of stationary-crystal diffraction patterns with changing temperature were taken using a single CTEP crystal at BL-1.

Faculty of Science and Engineering, Ritsumeikan,University

The preparation of the CTFP single crystal was the same as that used in the previous study [4]. The diffraction patterns were observed in thetemperature range from room temperature to 108 K. Figure 2 is stationary-crystal diffraction patterns of the CTFP with varying the temperature. The bottom pattern taken at room temperature of 183 K looks like a Laue pattern. There is a distinct difference between them, in that, it is possible to assign the photon energy to the spots, since the energy width is known. The spots of the bottom pattern are sharp, indicating that the incident beam is a parallel beam.

Figure 3 is an enlarged photograph of Fig. 2. It is clearly seen that a satellite reflections indexed by 0 k+1/5 l appeared below 158 K and the phase transition from the room temperature phase to a low temperature phase was confirmed. The most striking feature observed in the series of the diffraction patterns in Fig. 2 is that a reflection of satellite spots are somewhat diffuse compare with that of the Laue spots. Those reflections were never observation by a moving-crystal diffraction method using an ordinary laboratory X-ray diffractometer or camera[3]. Figure 4 shows a profile of the position and the intensity of the $0\bar{4}1$ Laue and its satellite spots observed at 123K. It is confirmed that a distance from the Laue spot to the satellite spots is kept constant for each temperature. On the other hand, a half width of the both spots is different. Both results indicate a nature of a superstructure structure and origin of the158 K phase transition. For a more accurate understanding of the phase transition of CTFP, the following studies are now in progress: all the observed spots of the half width of the both spots are now measuring. Thus, the system was successfully utilized for structural studies of phase transition.

REFERENCES

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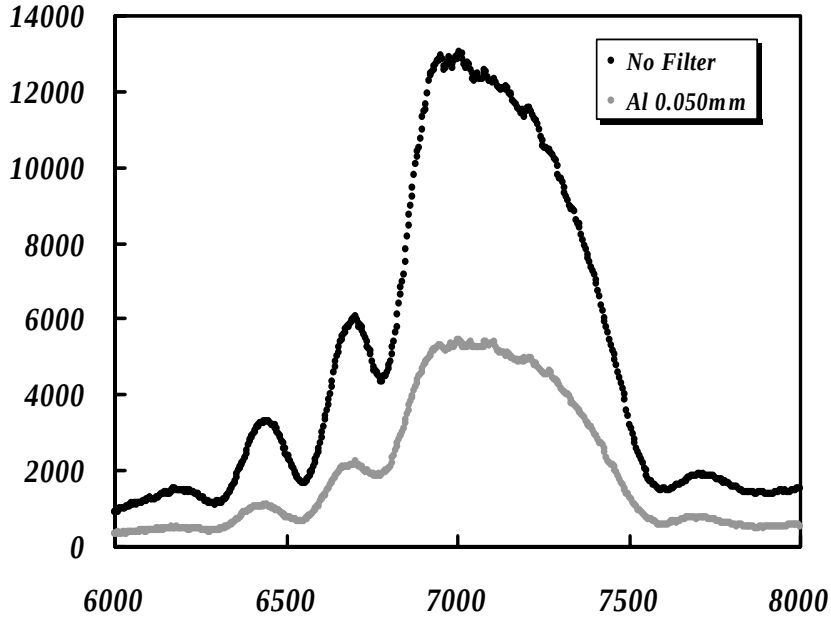

Fig.1. Energy spectrum of the beam reflected from the wide-bandpass multilayer monochromator.

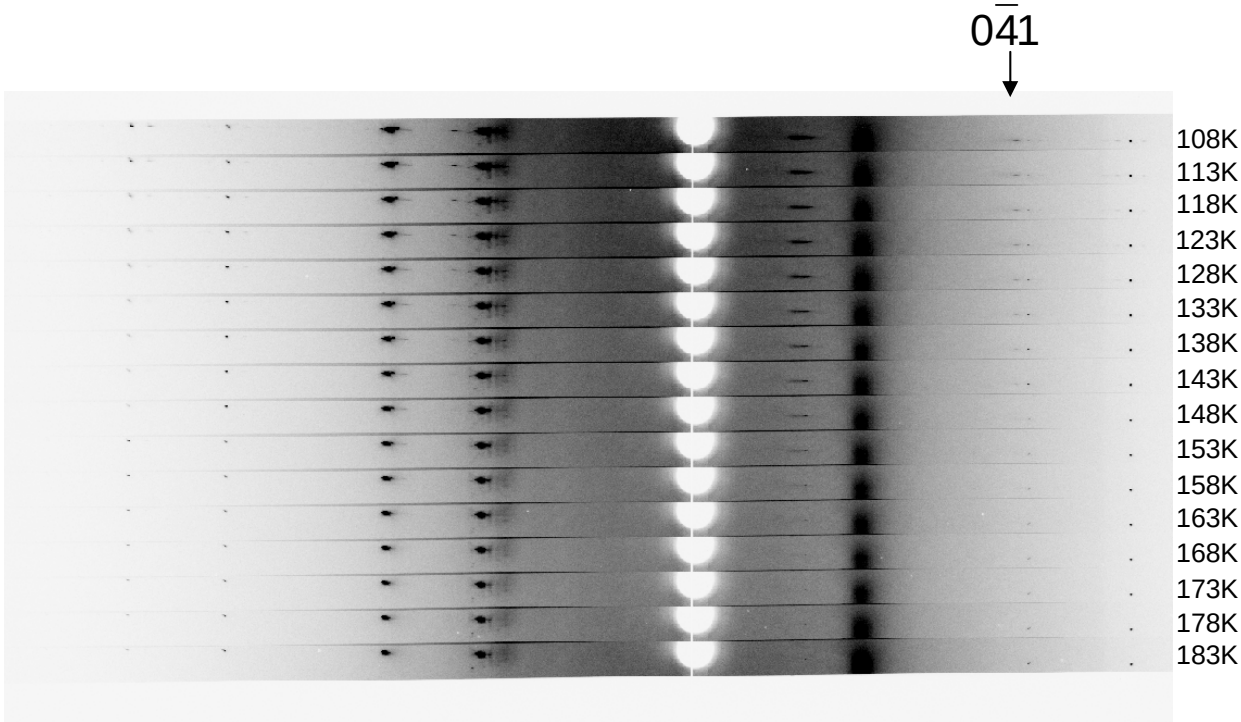

Fig. 2. Photograph illustrating the position of the Laue spots and the satellite spots with varying the temperature. Equatorial reflections 0kl from CTFP crystal. The diffraction patterns were observed in the temperature range from 183 to 113 K. Decreases by 5 K for each successive strip. The bottom strip is taken at room temperature phase of 183K.

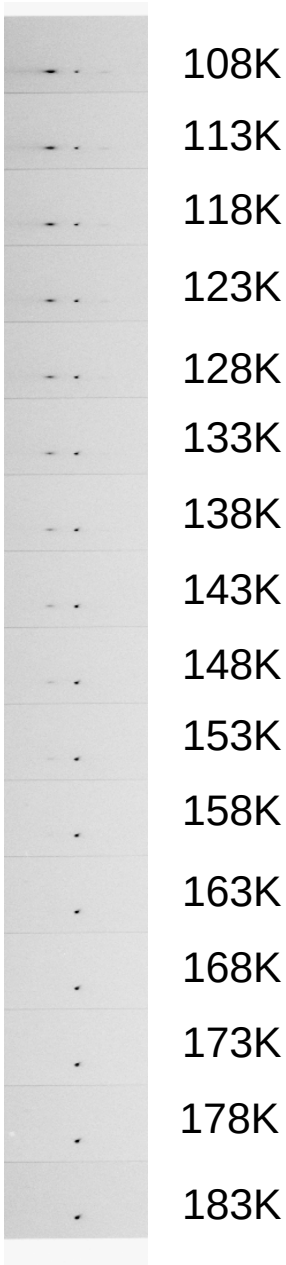

Fig. 3. An enlarged photograph of the Laue spots and the satellite spots in Fig. 2.

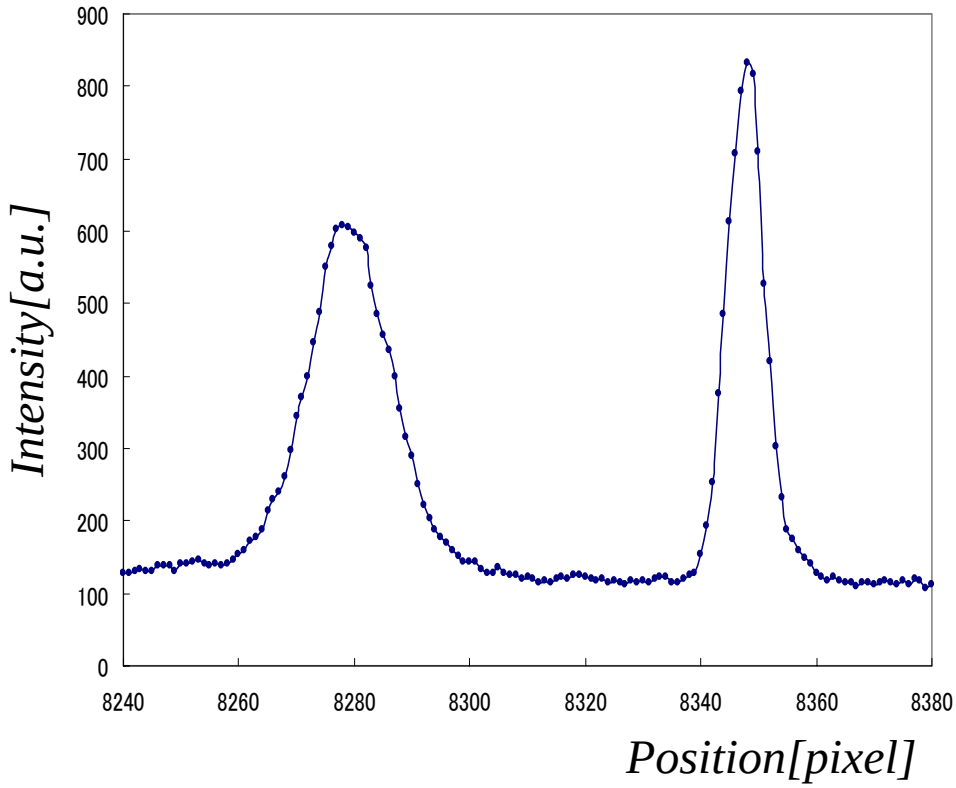

Fig.4. A profile of the position and the intensity of the $0\overline{4}1$ Laue and its satellite spot.