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Technical Research for High Quality X-ray Diffraction Measurement Using Open-flow Cryostat at BL14A

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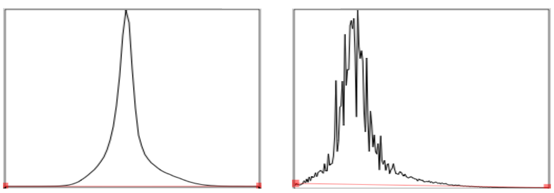
1 Introduction

Accuracy of diffracted intensity and high resolution are the most important two factors for quite accurate electron density measurement in crystals. In this regard, beamline 14A is the most sophisticated facilities. The beamline equips four-circle diffractometer installing avalanche photodiode detector (APD) whose counting loss is less than 1% up to 10^8 cps, and the four-circle diffractometer enables ψ-rotation which is effective to avoid contaminations due to multiple diffractions. Whereas the intrinsic qualification of this beam line is quite high, when open-flow cryogenic cooler is attached and the specimen is cooled, Blagg peak profiles are often splintered and integrated intensities are not correctly measured. The same problem is reported at German synchrotron beamline [1]. Since our aim is to determine the displacement of atoms which account for faint spontaneous polarization growing along *b*-axis in multiferroic YMn_2O_5 under ~40K, the splinter problem must be overcome. Therefore, we tried to investigate the cause of the problem squarely.

2 Experiment

At first, we have tried to detect the cause of splinter of Bragg peak profiles. Si single crystals were spherically grinded and mounted on three types of rod having different thermal conductivity; (A) sapphire rod, (B) borosilicate glass rod, and (C) sapphire rod but whose head is replaced with borosilicate glass rod within ~1mm using epoxy adhesive. These specimens are cooled to ~90K using open-flow cryogenic cooler, and Blagg peak profiles are observed.

The second test was aimed to find the countermeasure and evaluate its effect. Since the first test suggested that the thermal disconnection around the specimen from room temperature gasses can improve the shape of peak profiles, the borosilicate glass rod mounting the specimen on was coated by polyimide film remaining ~1mm from the top kept uncoated. Then, the specimen was cooled to ~23K using cold He gas flow and integrated intensities were collected using bisect setting and psi-rotated setting. Spherically shaped single crystal of YMn_2O_5 whose diameter is 245μm is used as a specimen, and 0.75392Å beam monochromated using Si111 double single crystal monochrometer is used as incident.



(a) (b)

Fig. 1: Bragg peak profiles blocking sheath gas (a) and without blocking (b) using polyimide film.

3 Results and Discussion

From the first test, broken Bragg peak profiles were observed for Specimen B and C, whose heads were both borosilicate glass and less conductive thermally. But for Specimen A, profiles were fine. After several other tests, we concluded that the thermal fluctuation near the boundary of the room temperature sheath gas and the cold He gas causes the expansion and contraction of borosilicate glass rod. Since we also confirmed that sapphire rod wormed by room temperature gases could heat the specimen unfavorably, we tried to block the room temperature sheath to hit the borosilicate glass rod. Figure 1 shows the result of before and after the polyimide film coating explained in the former section. Figure 2 shows the *Fobs-Fcalc* plots for bisect-setting and psi-rotated one. Even though, R-factor is still ~2.5% and still have room for improvement, we could solve the structure roughly in both measurements.

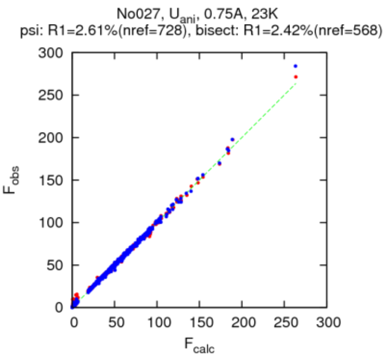


Fig. 2: Fobs-Fcalc plots for bisecting data collection (in red), and psi-rotated one (in blue).

References

[1] Y. Drees *et. al. DESY Photon Science Annual Report2010,* http://photon-science.desy.de/annual_report/files/2010/20101258.pdf

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