Crystallography

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DAFS and FRED on Satellite Diffraction of $Ca_2CoSi_2O_7$

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Introduction

Structural studies of synthetic Co-Åkermanite, $Ca_2CoSi_2O_7$, have been carried out by means of Mössbauer spectroscopy[1] and of X-ray diffraction[2] measurements. In the former study, indication of two distinguishable tetrahedral sites was found, and irrational superstructure diffractions were observed in the latter. These measurements indicate that the structure shows superlattice and/or incommensurate symmetry due to a long periodic modulation. Recently, Hagiya *et al.*[3] succeeded to determine the incommensurate structure of synthetic $Ca_2CoSi_2O_7$ by using the method of high dimensional structure determination. They concluded the followings: SiO_4 tetrahedron does not behave as a rigid body, the distance of Si-O tetrahedral bonds is variable throughout the crystal, Co-O distances are simultaneously and sinusoidally modulated. The results represent the satellite diffractions with incommensuratability δ=0.2913 to a+b and a-b directions, and moreover, they are well consistent with the results reported in Refs.[1, 2].

For detailed structure analysis, two problems were pointed out by Hagiya *et al.*[3]: one is that the higher harmonics of the satellite diffraction was not observed in their measurements, and another is that the superlattice diffraction was not taken into account for the structure determination. If in the case that the higher harmonics and the superlattice diffractions are considered, the mode of the incommensurate modulation and thus the structure model given in Ref.[3] must be required to be improved.

In order to solve the problems, we firstly aim to observe the intensity distribution in the reciprocal space, in particular at the points where the higher harmonics and superlattice diffractions are.

Experimental

The X-ray diffraction measurements were made on BL-10A beam line. The incident X-ray was tuned at 1.2 Å by 111 diffraction with silicon flat-plate monochromator. Intensity distribution was measured in the area -0.020<h<0.525, 0.980<k<1.520, l=0 with steps of $\Delta h=\Delta k=0.005$. Intensity was integrated for 5 sec. at each step by scintillation counter.

Results

The intensity map is shown in Fig.1. It is obvious that the higher harmonics satellites, for instance at (0.58 1.58 0.00), are clearly observed. This indicates that the mode of the incommensurate modulation proposed in Ref.[3] has

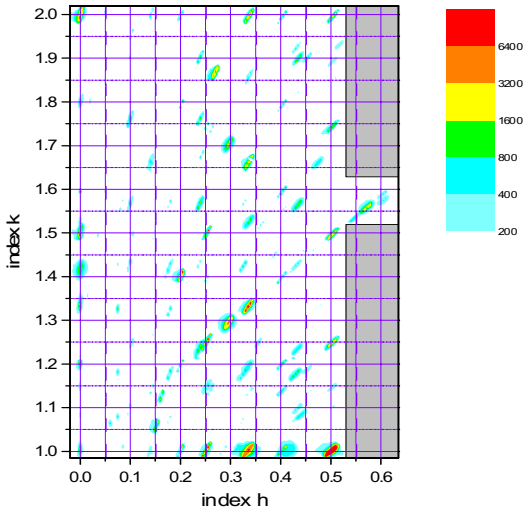

Fig.1 Intensity map on hk0 plane.

to be reinvestigated as pointed by the authors. In addition, it is noted that superlattice diffractions at many unexpected positions are simultaneously observed: for examples, at (0.20 1 0), (0.25 1 0), (0.33, 1 0) and (0.50 1 0). The superlattice structure which contributes such many additional iffractions has not been easily explained yet. For detailed analysis, we needs more complete intensity data in wider reciprocal lattice.

References

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