BL02B2: X-ray powder diffraction experiments and Rietveld analysis for precise structure determination

Abstract

Precise structure determination by Rietveld method using high-accurate powder diffraction data is now indispensable to materials science. In the BL practical, the participants will learn a standard procedure of synchrotron radiation powder diffraction experiments for obtaining highly accurate data, and technique for structural analysis with the Rietveld method. Advanced structural analysis for visualizing the electron density distribution conducted at BL02B2 will be demonstrated.

Practical Training

1. Selection of proper X-ray wavelength and capillary size

   In order to analyze the crystal structure from the powder diffraction data, it is important to collect accurate data without ambiguity. Here, we introduce how to select the proper X-ray wavelength and capillary size to obtain intensity and angular resolution without data correction.

2. Alignment of beamline components

   The optical system of BL02B2 is composed of two components, a collimator mirror for cutting higher harmonics and a double-crystal monochromator. In the beamline, alignment of the two components is carried out for the desired wavelength. The optical alignment is followed by alignment of the diffractometers,

such as a collimator, a goniometer and a direct beam stopper.

3. Preparation of powder sample

The accuracy of the powder diffraction data is influenced by grain size distribution of powder sample. A large grain results in inhomogeneity of intensity distribution on Debye-Sherrer rings. On the other hand, a small grain yields broadening of diffraction profiles. It is well known that the optimal particle size is a few micrometers. In the training, we will show you the precipitation method developed for easy filtering of powder sample and how to fill a capillary with powder sample.

4. Measurement of data using Imaging Plate (IP)

Using the aligned diffractometer, we will carefully measure the prepared sample. The capillary sample should be rotated during measurement to reduce an effect of grain size distribution and preferred orientation. The collected data is readout by the off-line IP reader outside the experimental hutch. First of all we should judge the quality of the powder sample from the 2-dimensional diffraction data. If the Debye-Scherrer rings have homogeneous distribution, the 2-dimensional diffraction data are converted to 1-dimensional diffraction data, where we should check the width of profiles. Finally we can start the data analysis.

5. Precise structural analysis by Rietveld method

Using accurate powder diffraction data, we lecture how to determine the crystal structure such as crystal system, Bravais lattice and space group *via* the program Fullprof Suite (http://www.ill.eu/sites/fullprof/). The basic process for the Rietveld

refinement with least-squares minimization between the observed and calculated profile will be mastered in the BL practice.