

Practical Training for Powder Diffraction Experiment at BL02B2

Abstract

The high-energy and high-brilliance synchrotron X-ray at SPring-8 allows materials scientists to unravel structure-property relationship with electron distribution resolution using powder diffractometry. In the present training, we are planning to offer practical technique on how to select proper X-ray wavelength and capillary size, how to align beamline components, how to prepare powder sample and how to measure data for synchrotron X-ray powder diffraction experiment. If time permits, the fully automatic measurement system using a sample changer and image recognition will be shown.

Practical Training

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

No ambiguity of obtained raw data is essential condition for accurate crystal structure analysis. Therefore, the beamline adopts transmission geometry using a curved 2-dimensional detector, Imaging Plate (IP). Here we introduce how to select the proper X-ray wavelength and capillary size taking account of diffraction intensity and angular resolution as well as no 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, input of the required wavelength allows us to align the two components.

The optical alignment is followed by alignment of the diffractometer, 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. The larger grain results in inhomogeneity of intensity distribution on Debye-Scherrer rings. On the other hand, the smaller 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 IP

Using the aligned diffractometer, we will measure the carefully prepared sample. The capillary sample should be oscillated over 360-degree rotation during measurement to reduce 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 data. If the Debye-Scherrer rings have homogeneous distribution, the 2-dimensional data are converted to 1-dimensional data, where we should check width of the profiles. Finally we can start the data analysis.