BL19B2 : X-ray Powder Diffraction Experiment

X-ray powder diffraction technique is a powerful tool for structural analysis of various materials. Utilization of synchrotron radiation to this technique ensures the efficient experiment for the researcher of new functional material because it realizes high-speed measurement and high quality data. In this practical training course, we plan to let you experience the measurement and the identification of unknown samples by means of X-ray powder diffraction using the large Debye-Scherrer camera with a sample changer at BL19B2. This course's purpose is to let XRD beginners know what kind of information about materials can be obtained from X-ray powder diffraction.

BL19B2 (Fig. 1) is a medium-length hard X-ray bending magnet beamline designed for engineering science researches. Target of this beamline is to

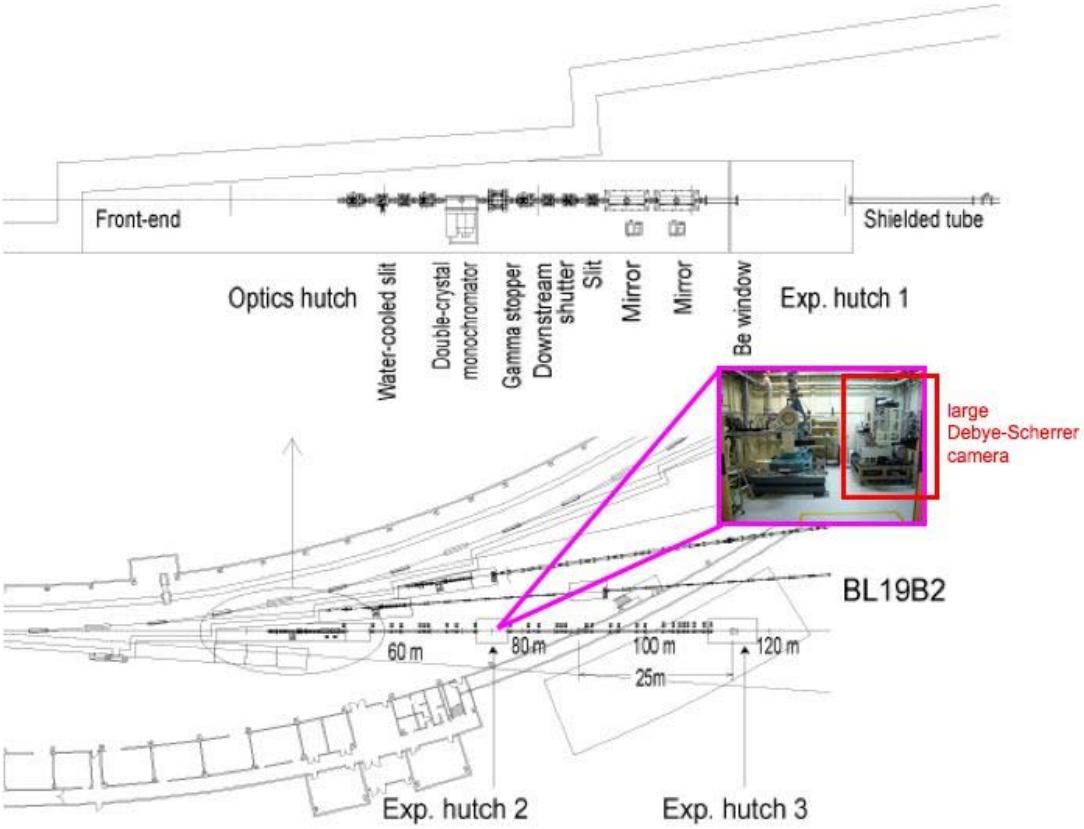

Fig. 1. Schematic layout of BL19B2.

promote the industrial application of the synchrotron radiation. The beamline has three experimental hutches for several kinds of experiments, including X-ray imaging, X-ray diffraction, and small-angle X-ray scattering. X-ray powder diffraction experiments are performed using the large Debye-Scherrer camera (Fig. 2) at the second experimental hutch. This camera was designed for accurate structure analysis. The basic specification of the large Debye-Scherrer camera installed at BL19B2 is the same as the one at BL02B2 of SPring-8. Imaging plates (IP) are used as 2-dimentional detectors. Users can change the sample temperature in the range of 90–1000 K by using a heater and a cryostat. These apparatuses utilize flowing nitrogen gas to control the sample temperature.

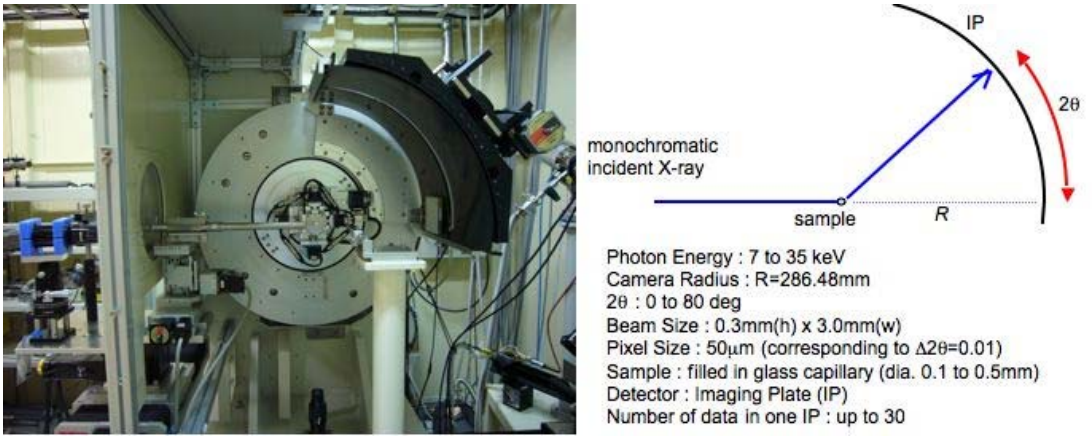

Fig. 2. The large Debye-Scherrer camera at BL19B2.

Industrial users need to screen a large number of samples for evaluating the quality of manufacturing or searching new functional materials. Therefore, high throughput is one of the important specifications required by them. In order to satisfy their requirement, we have developed an automated system, named “JukeBox” (Fig. 3), for synchrotron X-ray powder diffraction. The sample transfer robot and the automatic sample alignment system let users be free from cost of time and labor for exchanging and centering the samples. We utilize Microsoft Excel macros for the system control. This macro creates experimental condition files, and users can operate the automated system simply by filling cells with such information as sample

name, exposure time, and sample temperature. You might have no feeling of operation of the instruments.

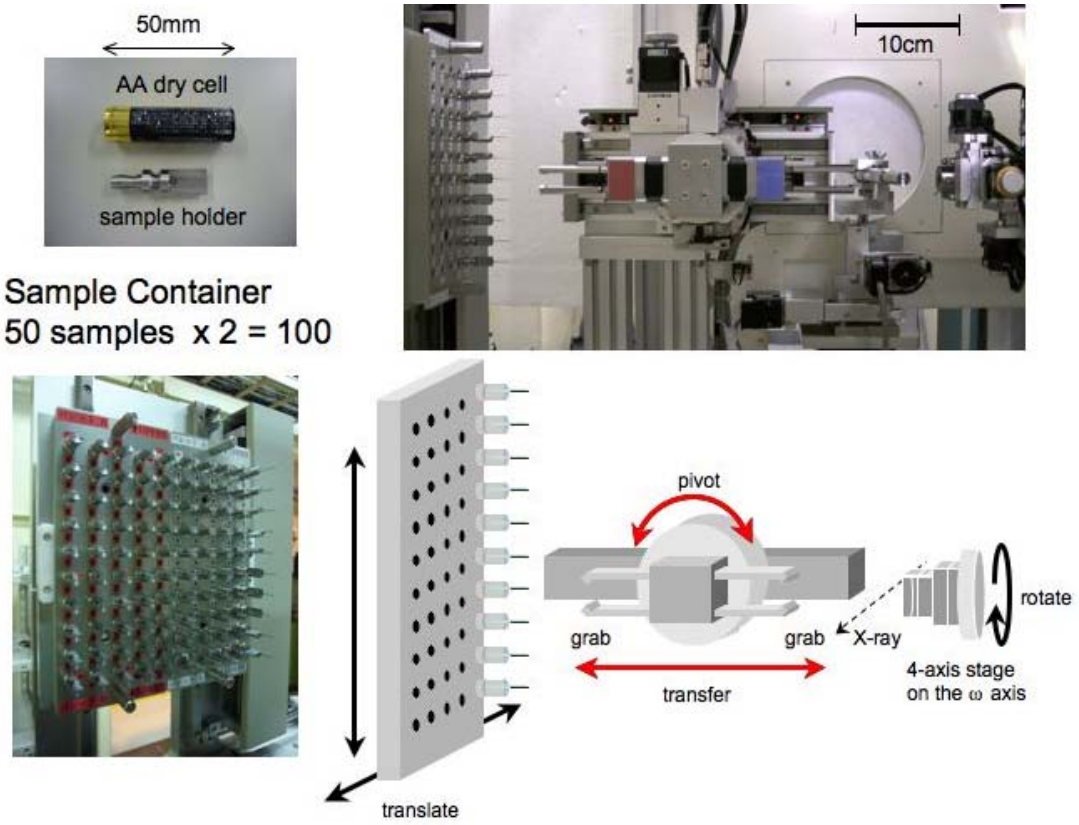

Fig. 3. The sample transfer robot in the automated system “JukeBox”.

You can learn in advance how to prepare powder samples and operate the large Debye-Scherrer camera by a brief video in the URL: http://support.spring8.or.jp/powder/jukebox.html. (Sorry, only in Japanese, but you should understand an overview of the experimental procedure.)

Practical menu is as follows:

1. Brief review of BL19B2 and the large Debye-Scherrer camera
2. Alignment of instruments
3. Preparation of powder samples
4. Data acquisition
5. Identification of the powder samples