High-Resolution Powder and Thin Film Diffraction

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1. Introduction

The powder method is very important for materials characterization in the fields of materials science and materials engineering. It handles I-dimensionally squeezed reciprocal data in data analysis. To obtain high-resolution powder diffraction data is, therefore, of primary importance for the *ab-initio* structure determination and accurate structure refinement. The use of parallel-beam synchrotron radiation can easily surpass a limitation in the angular resolution of laboratory systems .

Our objectives are, in the first, to evolve structure analysis techniques using high-resolution powder diffraction data and, secondly, their applications to structural studies of materials . There is a diversity in experimental setups in powder methods. SPring-8 will have beam lines for high pressure experiments using both white and monochromatic X-ray beams and for structural studies of phase transition phenomena. Thus the beam line could be specialized in high-resolution powder and thin film diffraction (HRPTFD) experiments.

2. Light Source

A bending magnet light source will be used for HRPTFD. It provides adequate beam sizes of l x l5mm at the sample position after tailoring. Most experiments are expected to use monochromatic X-ray beam in the angle-dispersive mode. Sharp monochromatic X-ray beams can be obtained by a single-step tuning of the Bragg angle of monochromator. Fast and precise tuning of energy is advantageous for successive measurements, in particular, in anomalous dispersion experiments . Linear polarization is suitable for the scanning in vertical plane using a flat or capillary specimen. The energy range will be expanded to 5~50 keV. The stability of beam intensity and beam position is important for powder diffraction scans, which usually take a half to one day.

3. Front End

Front end will consist of gate valves, photon-beam-position monitor (PBPM), masks with PBPM, absorbers, lead collimators, graphite filters, variable slits, beam shutter etc. A standardized front end will be used in the beam-line HRPTFD.

4. Optics

Main optical elements are a monochromator and a pair of mirrors. The water-cooled double-crystal monochromator will be installed. The height of the beam from the second crystal should be kept constant irrespective of the monochromator angle. One mirror on the upper stream of the monochromator is flat, and the other one on the downstream is of focusing-type. They are used to cut higher harmonics , to filter a low energy photons, and to reduce the thermal load for the remaining optics without changing the beam direction. Slit systems with water-cooled vertical and horizontal jaws will be set in front of the first mirror and the monochromator for defining the sizes of incident white beam.

5. Experimental Station

Fig. 1 shows a schematic diagram of powder diffractometer to be installed at the beam-line HRPTFD. The optics design for primary uses is based on a flat-specimen reflection geometry coupled with counter measurements . The multiple-detector system (MDS), which has been developed at the Photon Factory, will be used in order to resolve the problem of long scan time, accompanied with the 20-step scanning at a very small step size of, say, 0.001˚. It will have eight to ten counters, which are radially set at the interval of 20˚ to 15˚, and each of which will have Soller slits, an analyzer crystal, and a scintillation counter with counting efficiency

of more than 1M counts/s. A 2θ-scanning at a fixed incident angle will be used for measuring a whole-powder-pattern. The counter arm of the MDS at the highest angle can be used as a single-detector arm in either e-20 or 20-scan mode. The angular resolution of the instrumental function will range from 0.01˚ to 0.03˚ in ordinary diffraction experiments and to less than 0.01˚ for very high-resolution experiments . An X-ray camera using imaging plate can be attached at the lower half of the goniometer, and can be used in Debye-Scherrer geometry using a capillary containing a very small amount of specimen. A rotating flat or capillary specimen
can be used in the sample chamber, in which the air can be evacuated or replaced with gases. Beam sizes at the sample position (0.1 to 1mm in vertical direction and 1 to 20mm in horizontal direction) will be defined by using vertical and horizontal slits with stepper motors for positioning. The intensity decay of incident beam can be monitored by using both an ion chamber and a scintillation counter. High temperature furnaces and cryostats can be used to control the specimen temperature from Helium temperature to 2500˚C. The development of an universal specimen holder will be necessary for thin-film diffraction experiments. The diffractometer should be kept fixed at the experimental station to minimize the time required for instrumental alignment. Pulse-height analyzers will be automatically adjusted.

6. Data Processing

Computing systems will be based on the UNIX operating system. Output lines for the data from instrument-controlling system are to be connected with computers in a user's room for data analysis and further with house computers of individual users. Monitoring of data acquisition from remote facilities will become possible in a near future. Computer soft-wares for on-line data analysis will be available .

References

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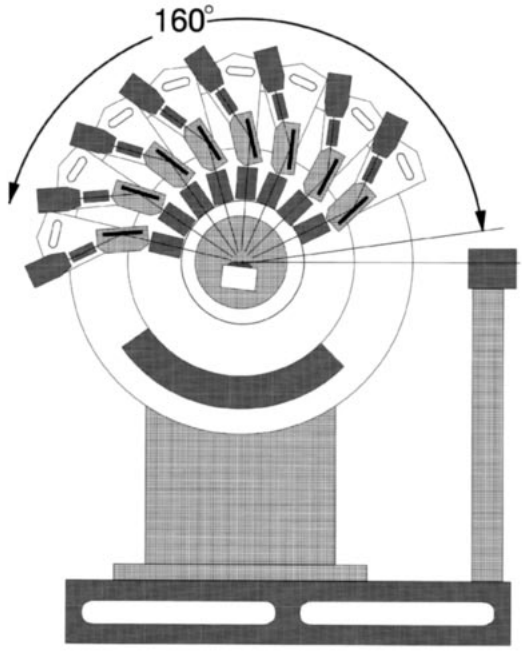

Fig. 1. A schematic diagram of MDS.