High-resolution Powder Diffraction Experiments at BL02B1

Hideo TORAYA
Makoto SAKATA
Masaki TAKATA

1. Introduction

Beam line BL02B1 is an experimental station for the structure analysis. It has a bending magnet light source, a pair of mirrors, a water cooled two-crystal monochromator, and a seven-axis diffractometer for multi-purpose experiments. Four subgroups (phase transition, powder diffraction, chemical reaction, and diffuse scattering) have been jointly working to construct the station. Experiments for testing the station were started from July 1997 (1st experiment). Experiments by the user group "powder diffraction" was conducted in December 1997 after facility was opened for public use at the beginning of October 1997 (2nd experiment). The present paper reports experimental results conducted during these two periods.

One of the aims of scientific researches at the Spring-8 for the powder group is the crystal structure analysis by the powder method using high-resolution diffraction data. Thus experimental time allowed for the powder group was devoted to examining the beam quality and the instrumental resolution using the diffractometer presently available at BL02B1.

2. Experiments

A flat-specimen reflection geometry was employed by using a rotating specimen of NIST SRM 640b Si powder or SRM 674a CeO2 powder, which was packed into a glass specimen holder. Crystal planes of monochromator used were Si(111) and Si(311), in which only the latter could be used in the 1st experiment. A second crystal of the monochromator was sagitally focused for converging the beam at a sample position in the 2nd experiment. The analyzer, mounted on a long detector arm of the diffractometer, was a Si(220) flat crystal or long parallel slits (LPS) with an aperture of 0.03˚. Mirrors were not used in these experiments. Ring current in the present experiments was typically around 19mA. A wavelength used was 0.5Å in the 1st experiment and it was 1.0Å in the 2nd.

3. Results and discussion

a) Analyzer crystal (0.5Å)

The monochromator, exposing a Si(311) plane to the incident white beam, was used to obtain a wavelength of 0.5 Å. On the diffracted beam side, an analyzer crystal (AC) of Si(220) was attached. Fig. 1 shows an observed diffraction profile of 422 reflection from CeO_2 powder. It was fitted with the pseudo-Voigt function, showing a well-resolved nearly symmetric profile shape.

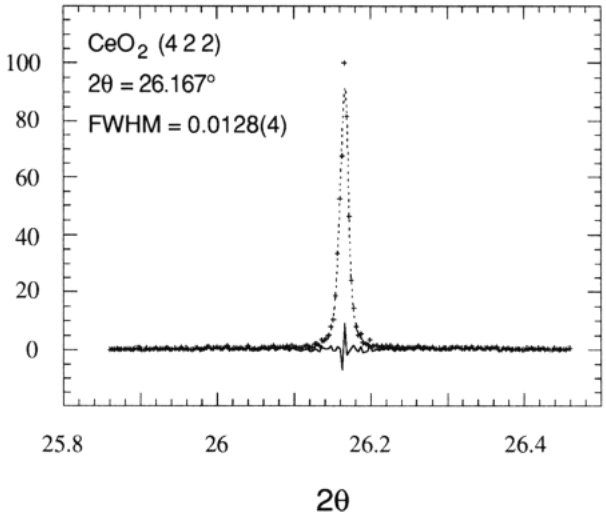

Fig1. Diffraction profile of 422 reflection from CeO_2 powder.

Fig. 2 shows variations of the FWHM's of Si and CeO_2 powders with 2q. The minimum FWH's were 0.0127˚ and 0.0136˚(2q) for CeO_2 and Si, respectively. The profile is not largely broadened with increasing 2q.

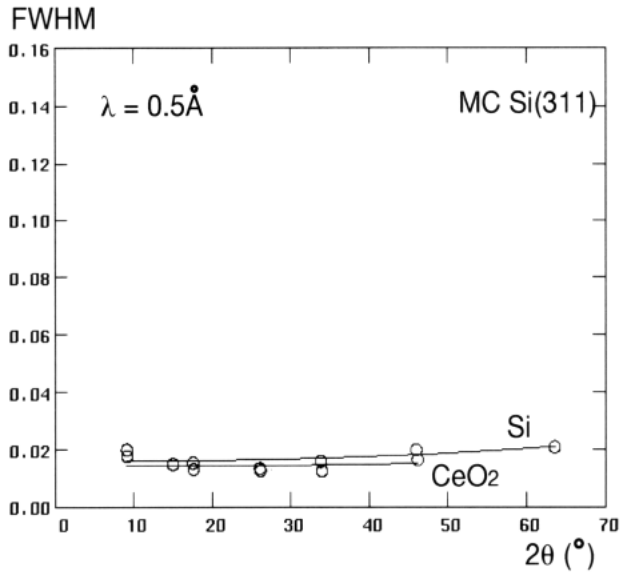

Fig. 2. Variations of FWHM's of diffraction profiles of Si and CeO_2 powders with 2q.

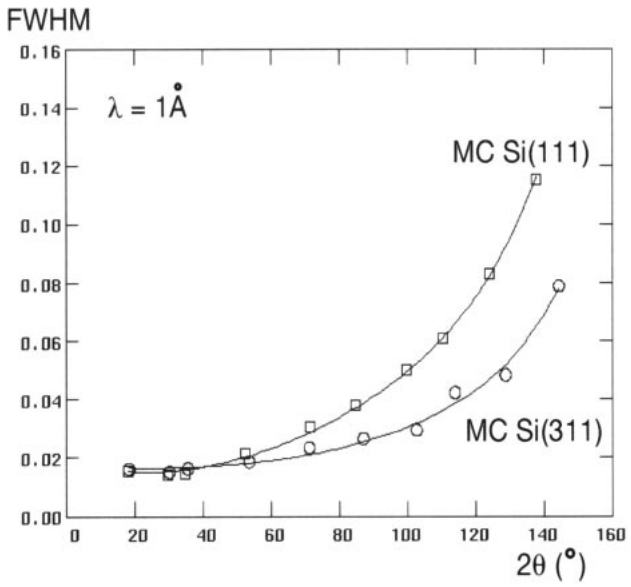

Fig. 3. Variations of FWHM's of diffraction profiles of Si powders with 2q. (MC: monochromator crystal)

b) Analyzer crystal (1Å)

Fig. 3 shows the variations of the FWHM's of Si powder with 2q for the wavelength of 1Å. Two results presented are obtained by using monochromator crystal (MC) of Si(111) + Si(220) AC and Si(311) MC + Si(220) AC. The FWHM was 0.014˚(2q) at the minimum for the former coupling and it was 0.015˚(2q) for the latter. There is little difference between the minimum FWHM's for the two couplings of MC and AC. The resolution was, however, much improved in the high-angle region if we used the latter coupling and sacrifice the intensity.

c) Long parallel-slits (0.5Å)

The minimum FWHM obtained with LPS using the beam with a wavelength of 0.5Å was 0.032˚ (2q) for 220 reflection from Si powder. The FWHM was 0.05˚ (2q) at 2q = 70˚. The observed resolution of LPS was well in accordance with a specification of the design of LPS. Diffraction profiles were, however, asymmetric, having high tails on both sides of the peak. Diffracted intensity from 111 reflection from Si powder exceeded 12kcps, and a much higher counting rate will be expected if the facility will be in full operation. The diffraction profile observed at the highest angular range was 13 71 reflection from Si powder.

Y. Kbota of Woman University of Osaka and H. Okudera, H. Hibino, M. Miyazaki, and H. Toraya of Nagoya Institute of Technology joined the high-resolution powder diffraction experiments, conducted during the period from July 1997 to December 1997.