High-Resolution Powder Diffraction Experiments at BL02B1

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1. Introduction

Powder diffraction experiments were conducted at the beam line BL02B1, which 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. The aim of the present experiments was to examine the beam quality and the instrumental resolution using the diffractometer presently available.

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(31l), 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. The observed diffraction profiles of reflections from CeO_2 powder show well-resolved nearly symmetric profile shapes. The minimum FWHM's were 0.0127˚ and 0.0136˚(2θ) for CeO_2 and Si, respectively. The profile is not largely broadened with increasing 2θ.

b) Analyzer crystal (1Å)

Two results were obtained by using monochromator crystal (MC) of Si(111) + Si(220) AC and Si(31l) MC + Si(220) AC. The FWHM was 0.014˚(2θ) at the minimum for the former coupling and it was 0.015˚(2θ) 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˚ (2θ) for 220 reflection from Si powder. The FWHM was 0.05˚ (2θ) at 2θ = 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 bc in full operation. The diffraction profile observed at the highest angular range was 13 71 reflection from Si powder.