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An eight-position capillary sample spinning stage for the diffractometer at BL20B at the Photon Factory

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An eight-position capillary sample spinning stage has been developed for use in conjunction with the versatile vacuum diffractometer (BIGDIFF) at BL20B at the Photon Factory. BIGDIFF is often used in its powder diffraction mode using powders mounted in capillaries and up to eight imaging plates to record the diffraction pattern from the sample. Using the multiple spinning stage a number of diffraction patterns can be recorded on the imaging plates if the imaging-plate cassette is moved behind the Weissenberg screen to a new position after exposure of the sample to the beam. Not only is this system more efficient in terms of time saved in the pumping-down process, but also it has the advantage of allowing the diffraction patterns of standards to be recorded, thereby calibrating both the angle scale of the diffractometer and the intensity scales of the imaging plates absolutely.

Keywords: capillary spinning stage; Weissenberg camera; imaging plates; powder diffraction.

1. Introduction

The Australian National Beamline at the Photon Factory is situated at bending magnet 20 of the Photon Factory storage ring, and its basic configuration has been described by Cookson *et al.* (1992) and Barnea *et al.* (1992). Two completely different monochromator configurations are available: a Hart-designed tunable channel-cut Si (111) monochromator (Cernik & Hart, 1989), and a separated-element fixed-exit-height monochromator with a Hart-designed first Si (111) crystal element (Berman & Hart, 1991) and a second Si (111) crystal mounted in a device designed to provide sagittal focusing (Stephens *et al.*, 1992). The sagittal focusing monochromator can give an increase in photon flux of 25 (Creagh & Garrett, 1995).

The principal experimental device at BL20B is a large vacuum diffractometer (BIGDIFF), which can be used in a variety of configurations. It can be configured to use imaging plates or diffracted beam monochromator and high-speed scintillator counters for photon detection, and either capillary or flat-plate sample holders. BIGDIFF is large (it has a radius of approximately 600 mm), and within its volume a wide variety of devices for tensometry (Creagh *et al.*, 1997), reflectometry (Foran *et al.*, 1998) and triple-axis diffraction can be mounted.

Its principal use is, however, for X-ray powder diffraction, especially for studies using imaging plates and capillary specimen holders. The performance of this powder diffractometer in the capillary mode has been described by Garrett *et al.* (1995), and its performance in both capillary and flat-plate mode was reported by Madsen *et al.* (1995). They showed that (i) its range of operation is much greater than that of most systems (<320°); (ii) it delivers a good undistorted lineshape over this angular range; (iii) for angles <70° the FWHM is almost constant at 0.05°, increasing to 0.25° at ±180° 2θ; (iv) exposure times for the imaging-plate mode can be very short (2 min) because all the diffraction lines are acquired at the same time; (v) scattered background is low because BIGDIFF is a vacuum diffractometer.

One of the problems with the fact that BIGDIFF is a vacuum diffractometer lies in the pumping-down time required (typically 20 min). This is usually much more than the exposure time and, from an operational point of view, an experimental sequence requires constant attention by the experimenter, which can be very fatiguing. In addition, angle positional error can occur because of lack of care in the positioning of the imaging plates.

This article describes equipment and an experimental strategy to overcome these deficiencies and provide an absolute calibration of position and intensity on the imaging plates.

2. Equipment

BIGDIFF was built with a facility for translation of the imaging-plate cassette as an integral part of the design. The entire cassette can be translated across the incident beam by the width of an imaging plate and, furthermore, the translation is carried out under computer control using the same *SPEC* software as used by all the motor/encoders in the BL20B system. In addition, a pair of Weissenberg screens can be fitted to BIGDIFF. One screen is bolted onto the diffractometer backplate and the other is mounted on the door of BIGDIFF. The resultant aperture is 5 mm.

A schematic diagram of the diffractometer and its Weissenberg slits is shown in Fig. 1. In this diagram the sagittal focusing monochromator is shown. The monochromator can be used in the unfocused mode, with the beam dimensions limited by primary and secondary monochromators upstream of the capillary. The capillary is mounted on a Huber 1005 goniometer head, which is coupled to a Huber 410/420 two-circle diffractometer system, and is at the centre of the imaging-plate camera system. X-rays scattered by the sample in Debye cones are prevented from illuminating the imaging plates except for the region of the gap between the Weissenberg screens, thereby restricting the length of the arc

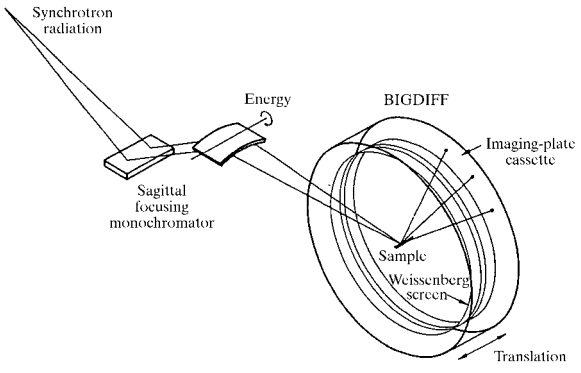

Figure 1
Schematic representation of the use of the BIGDIFF diffractometer in its imaging-plate mode using its Weissenberg screen. The imaging-plate cassette is translated across the incident beam by a linear stage driven by a stepping motor.

to ±2.5 mm with respect to the incident beam. It is possible to record up to 32 diffraction patterns on the same imaging plate.

This has significance for a number of applications in which a critical parameter is varied with time: for example, for DAFS (Creagh, 1995), in which the θ motion of the primary monochromator is linked to the motor that drives the imaging-plate cassette, or for multiwavelength anomalous dispersion (MAD) (Cookson *et al.*, 1998), in which measurements are taken at a number of discrete wavelengths.

In this instance, our concern was to reduce the time taken in pumping down BIGDIFF, as well as to provide the ability to record reference spectra. A special stage has been designed to assist in overcoming this problem. This stage carries eight Huber 1005 goniometers on which capillaries can be mounted. These are spun continuously, and each is brought into the beam under the control of a macro-instruction written for the *SPEC* software.

Each capillary is manually aligned on its goniometer head before mounting on the multicapillary spinner. It is then given a final manual adjustment using the TV camera system within BIGDIFF. The beam height is typically approximately 1000 µm, and measurement using counter techniques shows that the beam remains uniform over this distance. Given the positioning error of ±10 µm and the diameter of the capillary (50 µm), the specimen remains fully illuminated by the beam as it spins. For optimum accuracy, however, the capillary must be coaxial with the diffractometer axis. Fig. 2 is a photograph of the eight-position capillary changer mounted within BIGDIFF. This photograph gives a good indication of the size of the diffractometer. Of interest in the photograph are the imaging-plate clamps, which can be seen at the periphery of BIGDIFF.

The mechanism by which the capillary spinner operates is described below.

(*a*) An eight-position plate is bolted to the top of the Huber 410 (θ axis) of the two-circle diffractometer within BIGDIFF and the mounting plate is designed such that the axis of each of the goniometers will lie within ±10 µm of the diffractometer axis (Fig. 3*b*).

(*b*) The plate has mounts that interlink the rotating sample heads by using a toothed belt drive, which is driven by a 12 V DC motor at 220 r.p.m. (3.7 Hz).

(*c*) The eight heads are accurately indexed into the beam by a 45° Geneva mechanism (Fig. 3*a*). The Geneva mechanism moves when a pin in the driving wheel attached to a stepping motor engages one of the slots in the Geneva wheel. The motor is controlled by the main computer, which uses the *SPEC* program. When the pin exits from the slot after moving the wheel 45°, an arc segment on the driver engages a corresponding arc in the driven wheel, locking the mechanism in place. This mechanism overcomes the need to have a motor accurately position the samples as the interlocking arcs have a dwell angle of >180°. Error in the capillary position on change of goniometer is typically <10 µm.

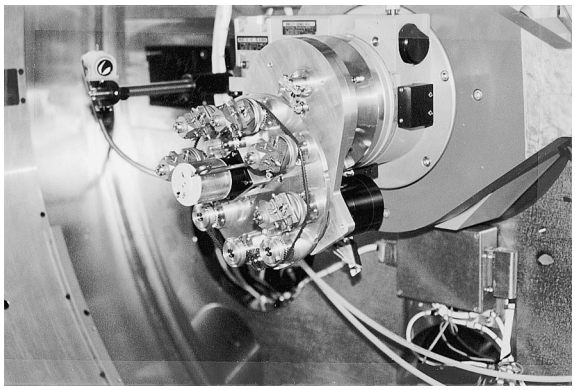

Figure 2
Photograph of the eight-position capillary spinning stage in position in BIGDIFF. Note the size of BIGDIFF.

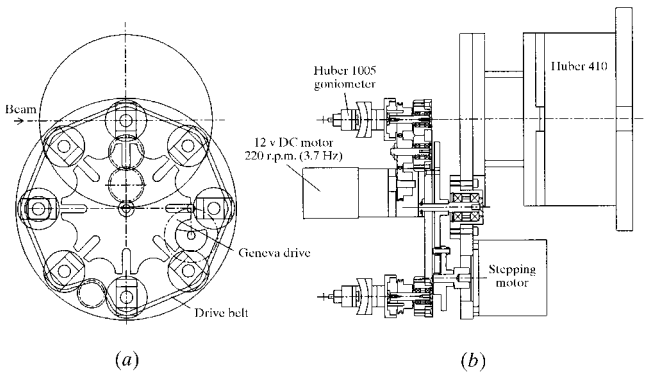

Figure 3
(*a*) Plan of the Geneva drive mechanism. The samples are constantly being spun at 3.7 Hz by a DC motor. When the stepping motor rotates through 360°, the plate that carries the goniometers rotates through 45° into the next fixed position. (*b*) Side view of the Geneva drive mechanism showing the location of the goniometer axis with the diffractometer axis, and the locations of the DC spinning motor and the Geneva drive's stepping motor.

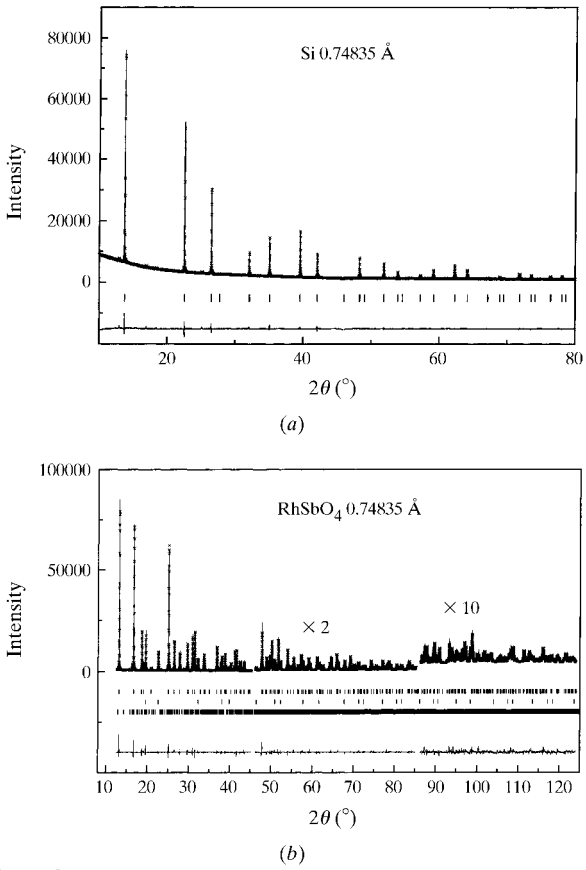

Figure 4
Observed (crosses), calculated and difference profiles for (*a*) silicon and (*b*) $RhSbO_4$ collected using the multiple sample holder. The short vertical markers show the presence of (from top to bottom) $RhSbO_4$, Rh and Rh_2O_3. The *R* factor is 0.086.

In an experiment, the imaging-plate cassette is set to its correct position with respect to the direction of the incident beam, and the standard sample is set into position. The imaging plates are exposed using the diffractometer beam shutter. The exposure time is determined by the scattering power of the standard chosen. After exposure, the cassette is moved through 10 mm, and the second goniometer is brought into position and exposed to the incident beam. This procedure is continued until the standard is brought into position. Another exposure is made, placing two diffraction patterns of the standard onto the imaging plates, one at the commencement of the experiment and the second at the end of the experiment. This provides a calibration of the decrement in incident beam intensity during the course of the experiment.

3. Results and conclusions

Because of the recent shutdown at the Photon Factory, only a limited number of experiments have been completed. Figs. 4(*a*) and 4(*b*) show data taken from an experimental sequence (Kennedy, 1997). Data were acquired at a wavelength of 0.74835 Å using only the upper segment (0–160°) of the imaging-plate camera. Fig. 4(*a*) shows data for an NBS standard silicon specimen, taken over 80° (two imaging plates). The Rietveld refinement difference functions are also shown. The peak positions and peak widths of the lines in the diffraction pattern are identical to those recorded by Garrett *et al.* (1995).

Fig. 4(*b*) shows the diffraction pattern taken for rhodium antimonate ($RhSbO_4$) in which impurity phases of free rhodium and rhodium oxide (Rh_2O_3) exist. The Rietveld refinement of this complex diffraction pattern was greatly assisted by the ability to determine the angular positions and absolute intensities of diffraction lines accurately because of the existence on the imaging plates of a standard spectrum. Refinement gave an R factor of 0.086.

The eight-position capillary spinning stage has proved that it can (*a*) provide the ability to record the diffraction patterns of standard materials and up to seven unknown materials, the standard material providing absolute calibration of angular position and intensities for the unknown materials, and (*b*) reduce very substantially time lost in pump-down and diffractometer supervision during a data-acquisition cycle.

When the Photon Factory recommences operation in November 1997, further experiments are to be undertaken to establish whether sagittal focusing causes any degradation of vertical resolution.

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