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COLLINS,, S. P., CERNIK, R. J., PATTISON, P., BELL, Anthony M. T. <http://orcid.org/0000-0001-5038-5621> and FITCH, A. N.

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A two-circle powder diffractometer for synchrotron radiation on Station 2.3 at the SRS

S. P. Collins, R. J. Cernik, and P. Pattison[a)]
Daresbury Laboratory, Warrington, WA4 4AD, United Kingdom

A. M. T. Bell and A. N. Fitch
Department of Chemistry, University of Keele, Keele, Staffordshire, United Kingdom

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The two-circle powder diffractometer on Station 8.3 at the SRS has recently been relocated to station 2.3, some half the distance from a dipole radiation source with the same nominal 1.2T field. The purpose of this paper is to detail the changes and modifications to the diffractometer operation.

The basic design and operating principles of the diffractometer remain unchanged, and are described in detail elsewhere.[1] Briefly, the instrument is based on the Parish–Hart design[2] and utilizes a large (~25-mm diameter) rotating flat-plate powder sample holder, with two sets of parallel foils on the detector arm to define the divergence of the diffracted beam. The primary long, fine foils are mounted perpendicular to the vertical diffraction plane and have a maximum angular acceptance of 0.07°. In addition, a set of coarser 1.2° foils are aligned parallel to the diffraction plane in order to reduce peak-shape asymmetries at low scattering angles. The theta (sample) and two-theta (detector) circles are controlled independently by encoder feedback systems with a specified accuracy of 0.3 mdeg on the two-theta circle.

The main modification to the diffractometer was the redesign of the theta circle support, enabling the detector arm to move through an extended range from $-120°$ to $+130°$. The most significant changes, however, are due to the reduction in source distance from 28 to 16 m. Although the predicted count-rate increase of a factor of 2.5–3 would have provided a significant improvement in the instrument performance, preliminary measurements gave unexpectedly high rates in excess of 200 000 cps from the NBS 640 Si 111 reflection at 1.5 Å. This rate, taken with a beam current of 200 mA and a germanium 111 monochromator, represents a sixfold increase over the original station. The explanation for this high-intensity increase is not clear, but it does lend support to the suggestion that the source tangent point for Station 8.3 may be far from the center of the bending magnet, in a region of significantly reduced magnetic field.

In order to test wavelength stability over a period of several electron beam injections, repeated scans were performed over the Si 333 powder reflection at $2\theta \sim 90°$. Changes in wavelength were therefore manifest as small peak centroid shifts that could be quantified precisely. Initial measurements with the noncooled Ge channel-cut monochromator from Station 8.3 exhibited shifts of up to 20 mdeg (corresponding to wavelength changes of ~3 parts in 10^4) which correlated almost perfectly with beam current variations. This effect was interpreted as arising from expansion of the monochromator crystal lattice with beam heating. To reduce these instabilities, a water-cooled Si 111 monochromator of the Hart design[3] was installed and the measurements repeated. Subsequent shifts were found to be reduced to 3–5 mdeg during any one beam fill, and up to 10 mdeg from one fill to the next. These drifts are consistent with known vertical source movements, indicating shifts of ~0.1–0.2 mm and 0.3–0.4 mm during and between injections, respectively.

It is worth noting that the effects of vertical source size and movements are reduced by the scattering geometry adopted for this instrument, whereby both the monochromator and sample diffract vertically. This is because the peak shift due to the change in wavelength when the source moves is partially offset by the opposing shift from the change in incident beam angle on the sample. The shift in 2θ powder peak position caused by a vertical angular source movement of $\Delta\theta_{\text{source}}$ can be written

$$\Delta(2\theta_{\text{sample}}) = \Delta\theta_{\text{source}}[2(\tan\theta_{\text{sample}}/\tan\theta_{\text{mono}}) \pm 1], \quad (1)$$

where the negative sign is appropriate for the present geometry, and positive for the opposite case. Clearly, the effects of beam movements are smaller at low angles in both instances, but significantly reduced with the current arrangement. Indeed, the effects of source movements should completely vanish when $2\tan\theta_{\text{sample}} = \tan\theta_{\text{mono}}$

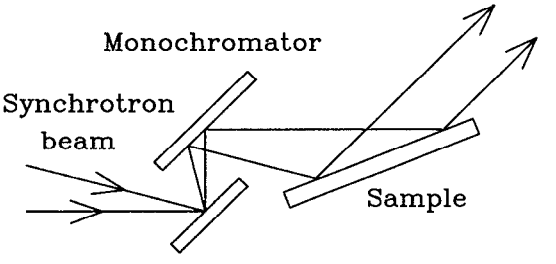

FIG. 1. A schematic diagram illustrating the insensitivity of the diffracted beam angle to vertical source movements when $2\tan\theta_{\text{sample}} \sim \tan\theta_{\text{mono}}$.

[a)]Present address: Institut de Cristallographie, Université de Lausanne, Lausanne, Switzerland.

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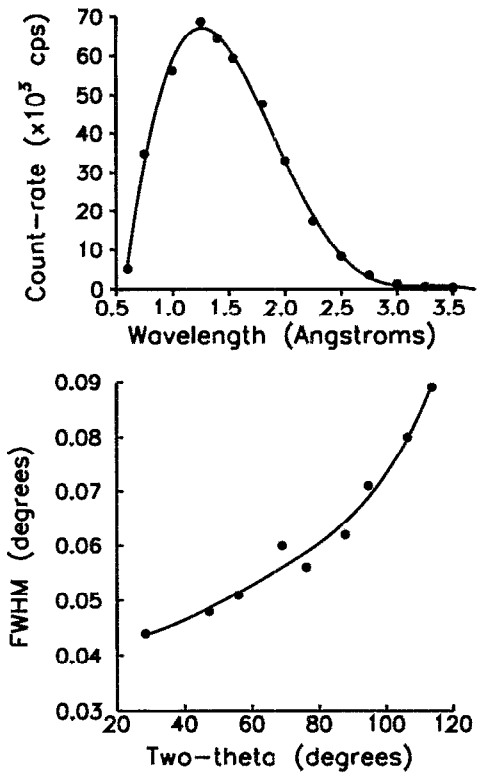

FIG. 2. The count-rates from the Si 111 powder peak as a function of wavelength (upper diagram). Data have been normalized to a slightly reduced beam current of 200 mA. The lower diagram shows the variation in half-widths for various Si powder lines with $\lambda \sim 1.5$ Å. It is assumed that particle-size and strain broadening effects in the sample are negligible.

(Fig. 1). In addition to improved stability, this setup should provide the best angular resolution.

To test these ideas, measurements were carried out at the Si 111 powder reflection (where $\theta_{\text{sample}} = \theta_{\text{mono}}$) and the resulting angular movements were found to be greatly reduced to less than 1 mdeg during any one beam fill. In addition, peak width measurements made by diffracting up (+ ve 2θ) and down (− ve 2θ) showed a clear reduction in the half-widths for the + ve scans of around 10% over the entire range of scattering angles, providing further support for this picture. It is therefore clearly advantageous to diffract up (or down) with both the monochromator and sample with this type of instrument.

The narrower bandpass of the new Si 111 monochromator has produced both an improvement in resolution of around 10% and a reduction in intensity by a factor of ~ 2.5. However, the count rates are still doubled compared with Station 8.3, and the diffractometer operation has therefore been improved in both count rate and resolution (see Fig. 2). A water-cooled Ge monochromator is planned for the station for future use.

In addition to the high-resolution ambient studies, the diffractometer can be fitted with a low-temperature sample stage, based on an Oxford Instruments continuous-flow helium cryostat. Rotating flat-plate sample holders are again used, and can be maintained at temperatures between 10–300 K. Sample temperatures are measured with a RhFe resistance thermometer, and have been found to be stable to a few tenths of a degree, and accurate to within 1 K.

The simplicity of this type of diffractometer, coupled with the inherent insensitivity to sample misalignment, have lead to highly efficient operation at ambient and low temperatures. With the increased count rates and faster software now available, several samples can be measured in a day, with typical scan times of a few hours.

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