High-speed, Compact Capillary Spinner for Powder Diffraction

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Introduction

The undulator beamlines at the APS are excellent tools for high-resolution powder diffraction [1] and can be used to obtain data on small quantities of samples in a short period of time (less than 0.5 second per data point in a continuous scan). It is often convenient to take data in transmission mode, with the sample sealed in a glass capillary with a diameter that is between 0.7 and 2 mm. Because of the high collimation of the undulator beam and the small volume of sample that is illuminated in the capillary, diffraction line profiles are dominated by speckle unless the sample is rotated continuously during data acquisition. To minimize the speckle, a good rule of thumb is to perform a complete rotation of the sample for each data point. Because a typical rotation stage can be moved at several degrees per second, the lower bound on the data collection time per point is well over a minute when this guideline is used. Typical fluxes at an undulator such as that at MR-CAT Sector 10 at the APS make it wasteful to collect data on this timescale when sufficient counting statistics can be obtained 100 times more rapidly. Our solution to this problem is a high-speed capillary spinner made with a low-cost motor and a custom housing that can be attached directly to a large IUCr goniometer mount. This spinner is shown in Fig. 1.

Methods and Materials

The spinner was constructed from a Radio Shack Model 273-255 12-V dc motor mounted in a custom aluminum housing. This motor has a variable speed of up to 15,000 rpm, depending on the load and applied voltage. For most applications, a minimum voltage of 1.4 V is sufficient to rotate the capillary at approximately 200 rpm. The capillary is mounted onto the motor shaft by using a 1-cm-diameter aluminum collett that is 1.5 cm in length with three set screws on the shaft side and a hole having a diameter that is slightly larger than the diameter of the capillary being

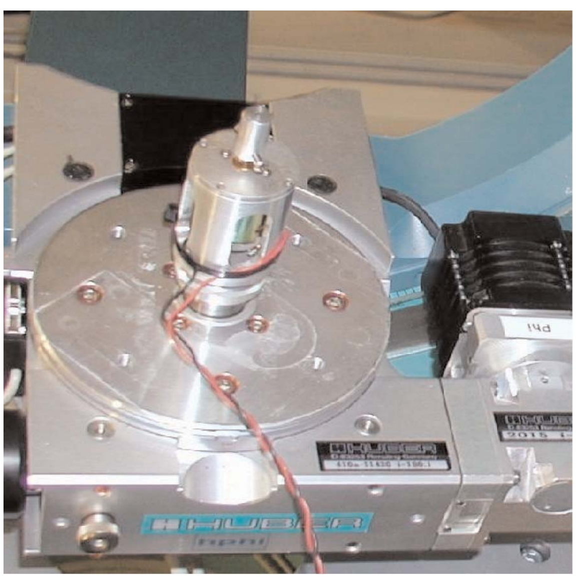

FIG 1. Capillary spinner mounted on a Huber 410 rotation stage with a large IUCr mount. The capillary (not present in this figure) is mounted in the cylindrical collett attached to the shaft of the motor and held in place with modeling clay. The rotation axis of the capillary is trued during rotation by applying a gentle pressure on either side.

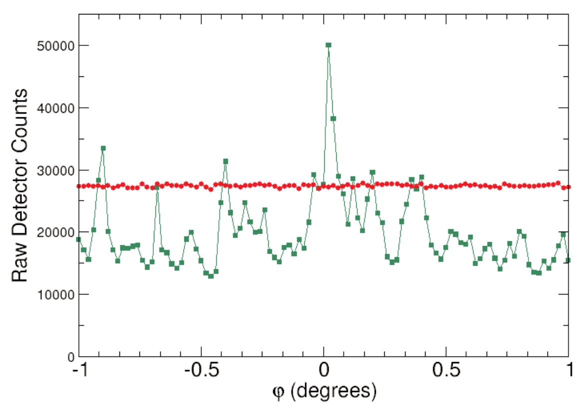

FIG 2. Effect of capillary spinner on the $2\theta = 9.647°$ diffraction peak of $(Ba_{0.7}Sr_{1.3})TiO_4$ at an x-ray wavelength λ of 0.5 Å. The data were collected in transmission through a 0.7-mm capillary with a flat Si(111) analyzer crystal in front of the scintillation detector. The storage ring was in top-up mode, giving a constant incident intensity to within 0.5%. The data shown in green were obtained by rocking the capillary through an angle of 2° with a collection time of 1 second per data point. The data shown in red were taken by using the identical rocking motion and collection time, but the sample spinner was turned on. The error bars fall within the symbols.

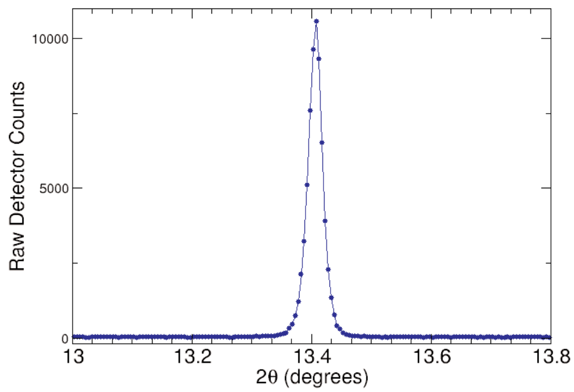

FIG. 3. A segment of powder diffraction data taken on a 1-mm capillary of MgB_2. Conditions are identical to those in Fig. 2, except the data collection time per point is 0.1 second.

used. The capillary is held in the collett by modeling clay, and its rotation axis is trued by applying gentle pressure with the fingers on either side of the glass while the motor is rotating at low speed.

To demonstrate the effectiveness of the spinner in reducing speckle during rapid data collection, data were taken when the capillary spinner was used, as shown in Fig. 2. The spinner clearly removed the effects of speckle at a 1-second data acquisition time.

Results

Figure 3 shows a small fragment of data collected on a 1-mm capillary of MgB_2 by using the spinner and a data acquisition time of 0.1 second per point. The data show an excellent signal-to-noise ratio out to high angles, with no evidence of speckle.

Discussion

This compact, low-cost capillary spinner has been shown to be effective in removing speckle from powder diffraction data collected at the high speeds permitted by an APS undulator.

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References

[1] C.U. Segre, N.E. Leyarovska, L.D. Chapman, W.M. Lavender, P. Plag, A.S. King, A.J. Kropf, B.A. Bunker, K.M. Kemner, P. Dutta, R.S. Duran, and J. Kaduk, *Synchrotron Radiation Instrumentation: Eleventh U.S. Conference* **CP521**, 419-422 (AIP, New York, NY, 2000).