	Experiment title: Further development of a method of crystal structure determination using transmission powder diffraction data from textured samples	Experiment number: MI-482
Beamline: BM01A	Date of experiment: from: 23-Mar-01 to: 27-3-01 13-Jul-01 17-Jul-01	Date of report: 7-Feb-02
Shifts: 18	Local contact(s): Jon Are Beukes	*Received at ESRF:*

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Report:

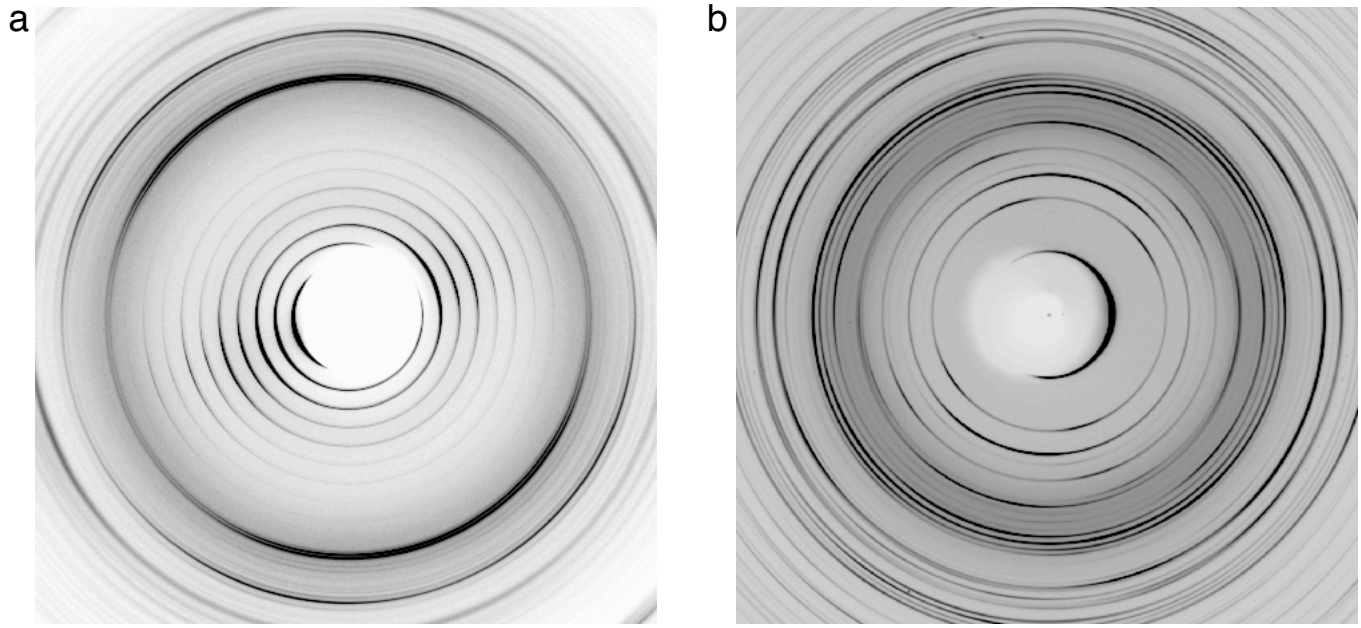
These experiments were part of a larger project devoted to the development of a method of crystal structure determination using transmission powder diffraction data from textured samples, and involved (1) a number of test measurements that were necessary for the development of the data analysis software, (2) an evaluation of various data collection strategies, and (3) the collection of full data sets on several samples whose structures are unknown.

Untextured samples of a Si standard and of zeolite A were measured for calibration purposes and to investigate a problem that had been encountered with the analysis of the image plate data. In our data analysis, 72 powder diffraction patterns, corresponding to 5° radial wedges are generated from each image plate frame using the program *Fit2d*. Previous analysis had shown that the peak positions were not completely consistent from wedge to wedge, and since this is critical to our subsequent data analysis, the cause of the discrepancy had to be investigated and a solution to the problem found. Consequently, some untextured samples, which should yield a series of identical patterns,

were measured. After a long evaluation process, we have come to the conclusion that the discrepancy probably lies in the readout routine of the image plate itself, and we are currently working on implementing an empirical correction to the data.

A full data set consists of 36 frames (each corresponding to a 5° rotation of the textured sample), so 36x72=2592 powder diffraction patterns have to be evaluated. To deal with this large amount of data, the data analysis software had to be streamlined so that integrated intensities could be extracted automatically. That software development is now almost complete. Once the empirical correction mentioned above has been implemented, analysis of the data collected on real samples can be started.

Of the samples examined, the most promising data were collected on the microporous aluminophosphate AlPO-M, silver behenate (used in photographic films) and Dodecanoyl-N-methylglucamide. The structures of these materials are not presently known and could not be determined from conventional powder diffraction data, so each dataset is expected to provide a challenge to the texture method. Examples of the data are given in the figures below.



Examples of data collected on well-textured samples showing smooth *hkl*-dependent variations in intensity around the rings for (a) silver behenate and (b) AlPO-M.

The analysis of these three datasets will begin as soon as the software is ready.