

**Experiment title:**

Development of a method for crystal structure determination using transmission powder diffraction data from textured samples

Experiment number:
01-02-293

Beamline:

BM01A

Date of experiment:

from: 2-Sep-00 to: 5-Sep-00
5-Oct-00

Date of report:

13-Dec-01

Shifts:

12

Local contact(s):

Hermann Emerich

Received at UNIL:

Names and affiliations of applicants (* indicates experimentalists):

*Christian Bärlocher, Lab. für Kristallographie, ETHZ, Zürich

Lynne McCusker, Lab. für Kristallographie, ETHZ, Zürich

*Thomas Wessels, Lab. für Kristallographie, ETHZ, Zürich

Report:

During the course of the last two years, we have been investigating the feasibility of transferring the data collection for our texture approach to structure determination [1,2] from a reflection to a transmission geometry. The first experiments (MI-385) indicated that there would be significant advantages in making this transfer (reduction of beamtime required per sample from 3 days to 3-6 hours, more complete data) despite the fact that some resolution in 2θ would be lost. The next two visits to SNBL (this experiment) were used to investigate various practical aspects of the data collection strategy and sample preparation.

In particular, several samples of the high-silica zeolite ZSM-5 with different crystallite sizes were examined to establish the influence of "graininess" on the data analysis, to determine just how small a sample could be used, and to investigate the possibility of applying the texture approach to small (roughly aligned) agglomerates of microcrystals. To test the limits of the latter, some protein crystal samples were also measured. During the second visit these were re-examined with the cryostream in place.

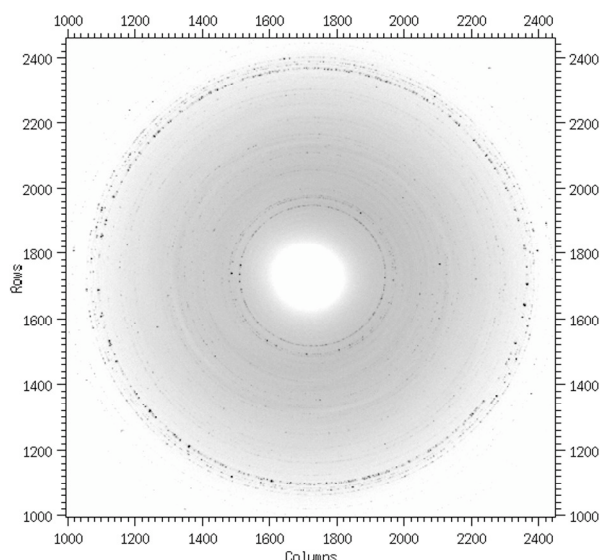


Figure 1. One image plate frame collected on a very small sample of ZSM-5.

The ZSM-5 results showed that data can in fact be collected on extremely small samples (Figure 1). The sample used to produce the image in Figure 1 consisted of just a few crystallites mounted on the end of a glass fiber. No attempt was made to orient them. The idea is that the "grainy" data be handled as one would a sample with a complicated, but very sharp, texture. This approach could be useful in cases where single microcrystals cannot be measured.

If an agglomerate of crystallites should display a natural orientation, as is often the case in polycrystalline materials, the data analysis should be even more straightforward. To test this idea and to challenge the limits of this approach to structure determination, data were also collected on a small fan of protein

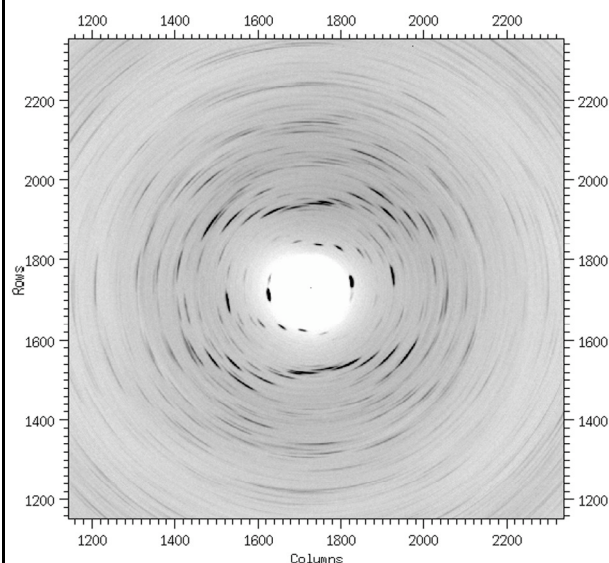


Figure 2. One image plate frame collected on a small fan of protein crystallites.

crystallites (Figure 2). A very strong texture is readily apparent, and the separation of reflections that would normally overlap in a diffraction pattern are clearly separated in orientation space.

In order to perform the data analysis, the software written for reflection geometry has to be adapted to the huge amount of data inherent to the transmission experiment. A typical data set consists of 36 frames (each corresponding to a 5° rotation of the sample, \square), and each frame is divided into 72 radial sectors (corresponding to 5° of sample tilt, \square). This means $36 \times 72 = 2592$ diffraction patterns (each corresponding to a different sample orientation (\square, \square)) must be treated. That adaptation is in progress.

References

- [1] Wessels, T., Baerlocher, Ch. & McCusker, L.B., *Science*, **284**, 477-479 (1999).
- [2] McCusker, Baerlocher, Ch., Grosse-Kunstleve, R., Brenner, S. and Wessels, T., *Chimia*, **55**, 497-504 (2001).