Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Development of a method for crystal structure determination using transmission powder diffraction data from textured samples	**Experiment number**: MI-385
Beamline: BM01A	**Date of experiment**: from: 29.3.00 to: 31.3.00; 1.6.00 to 3.6.00; 21.7.00 to 25.7.00	**Date of report**: 1-Sep-00
Shifts: 3x6=18	**Local contact(s)**: Hermann Emerich	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Christian Baerlocher

*Lynne McCusker

*Thomas Wessels

*Sinisa Prokic

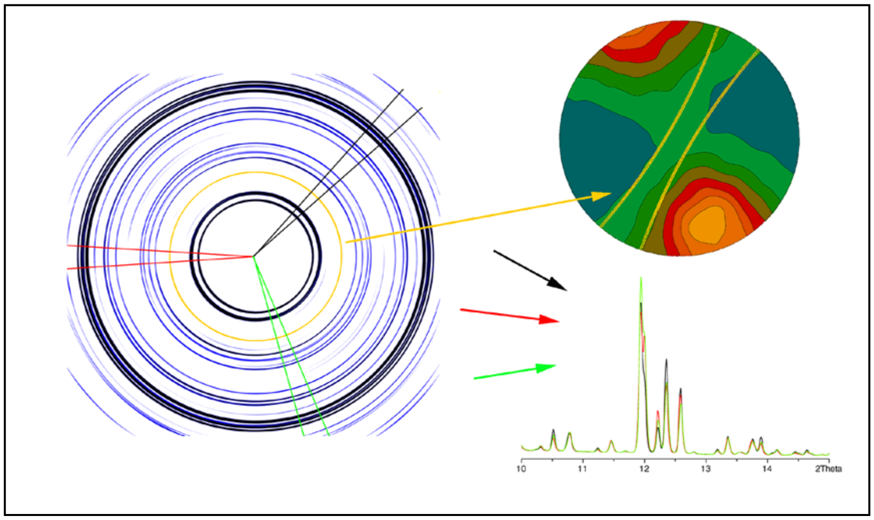
Laboratory of Crystallography, ETH, Zurich, Switzerland

Report:

In previous work at the ESRF, a method of structure determination for polycrystalline materials that exploits an intentionally induced preferred orientation of the crystallites was developed. Those experiments were performed in reflection mode, and were very successful. However, they required approximately 3 days of synchrotron beamtime per sample. It was hoped that the amount of beamtime required could be reduced significantly by performing the experiment in transmission mode and using an image plate detector system. The exploration of this possibility was the purpose of this experiment. To make optimum use of the allocated beamtime, it was divided into three blocks of six shifts. This allowed us to learn from one experiment in the planning of the next, and the strategy has proven to be an effective one.

The first of the three experiments was carried out in March. Initial measurements were performed to establish the optimal sample size, sample-to-detector distance and detector pixel size. For these measurements, small sections cut from a textured sample of the zeolite ZSM-5 were used. The results were not surprising. It quickly became clear that (1) the sample must be smaller than the focused beam (0.3mm) in all orientations, (2) the 2θ resolution of the diffraction pattern is significantly better for the maximum 400 mm sample-to-detector distance, (3) in order to access lower *d*-spacings (i.e. higher 2θ angle), a second measurement with a shorter sample-to-detector distance should be performed, and (4) a 100 µm rather than a 150 µm pixel size yields a better resolution of the peaks and this should not be sacrificed for data storage considerations.

check the consistency between the 0-180° and the 180-360° ranges. This highlighted the problem of sample inhomogeneity. Another set of redundant measurements were performed with the sample turned 90° with respect to the beam. The evaluation of these data showed the data to be consistent, but the analysis of some regions was complicated by the unavoidable presence of a shadow of the sample orientation device on the detector. A representative image of the ZSM-5 sample (left), a pole figure calculated from these data (top right), and diffraction patterns for the three different sample orientations indicated in the image (bottom right) is shown in the figure on the left.



For the second set of measurements, performed in June, a series of fresh samples were prepared. The information from the first series of experiments could be used to optimize the data collection strategy, and some full data sets were measured for the further development of the data analysis programs. Unfortunately, during the data analysis it became apparent that the glue used to attach the sample to the goniometer head contained a polycrystalline phase and the data were contaminated with a number of additional diffraction lines.

In the third experiment in July, the image plate was raised by approximately 10cm so that higher angle data could be collected with a longer sample-to-detector distance. Measurements were performed with the detector 180 and 300 mm from the sample, so that full circles could still be recorded for the low angle reflections. To avoid problems of detector saturation for the stronger reflections, two exposures of ca 200s each were performed for each sample orientation and detector position.

Analysis of these data is still in progress, but it is already apparent that the transmission geometry offers a number of advantages over the reflection setup. Not only does it appear to be possible to collect a full set of data in less than 6 hours (compared with 3 days for the reflection experiment), there are certain aspects of the data quality that are of particular importance in the data analysis. For example, (1) the intensity statistics at high angle are better with the area detector, (2) no correction for the sample tilt angle is required, (3) full pole figure data are intrinsic to the data collection strategy and this improves the determination of the texture of the sample, (4) partially overlapping reflections can be used for the texture analysis because the area detector provides all the necessary information (i.e. the full profile of the rings rather than the intensity variation for a just single 2θ value), and (5) full diffraction patterns for all sample orientations are measured. The latter should improve the subsequent extraction of individual reflection intensities. These advantages more than compensate for the loss of angular resolution inherent to the use of an image plate.