ESRF	**Experiment title: Further deveopment of a method of structure determination using transmission powder diffraction data collected on textured polycrystalline samples**	**Experiment number**: CH-1526
Beamline: BM01A	**Date of experiment**: from: 5-Dec-2003 to: 9-Dec-2003	**Date of report**: 11-Feb-2005
Shifts: 12	**Local contact(s)**: Philip Pattison	*Received at ESRF:*

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Report:

This experiment is part of a larger project devoted to the development of a method to unravel reflections that overlap in a powder diffraction pattern by exploiting preferred orientation. Previous experiments performed in transmission mode with a 0.2-0.3 mm textured sample and an imaging plate (MI-385, 01-02-293, MI-482, CH-1318) had shown that the resolution needed to be improved if more complex structures were to be tackled. Consequently, the sample-to-detector distance was set to a maximum (ca 400mm) to obtain the best possible peakwidths (ca 0.05˚2θ), and the detector was displaced diagonally with respect to the primary beam to maximize the range in 2θ ($2\theta_{max}$~30˚).

Data were collected on many different samples using this setup. Some of these had been measured in previous experiments with the imaging plate centered on the beam, some had known structures, some had been prepared using different methods for orienting the crystallites, and some had not been measured before and had unknown structures. A single data collection consists of 72 imaging plate exposures, each corresponding to a sample rotation (ψ) of 5˚. Each imaging plate frame is then divided into nineteen radial wedges, each corresponding to a 5˚ sample tilt (δ), and thus the diffraction patterns for 72x19=1368 (1296 unique) sample orientations (ψ,δ) are generated (using the program *Fit2d* [1]).

Not all of the datasets have been analyzed yet, but those that have include mica oriented in polyethylene, the aluminophosphate $AlPO_4$-M, the potassium calcium silicate CAS-1, and the silicate E-401. The first was investigated to establish how well the mica particles in different parts of the sample were aligned after being subjected to different treatments, and

that analysis has been published [2]. The next two were control samples that had been studied (and their structures solved [3,4]) in previous experiments. Comparison of the data from the new setup with those from the old one showed the expected improvement in resolution (both in peak width and in 2θ range). A typical imaging plate frame from E-401, whose structure is unknown, is shown in Figure 1. The presence of a preferred orientation of the crystallites is readily apparent from the fluctuations in the intensity around the rings.

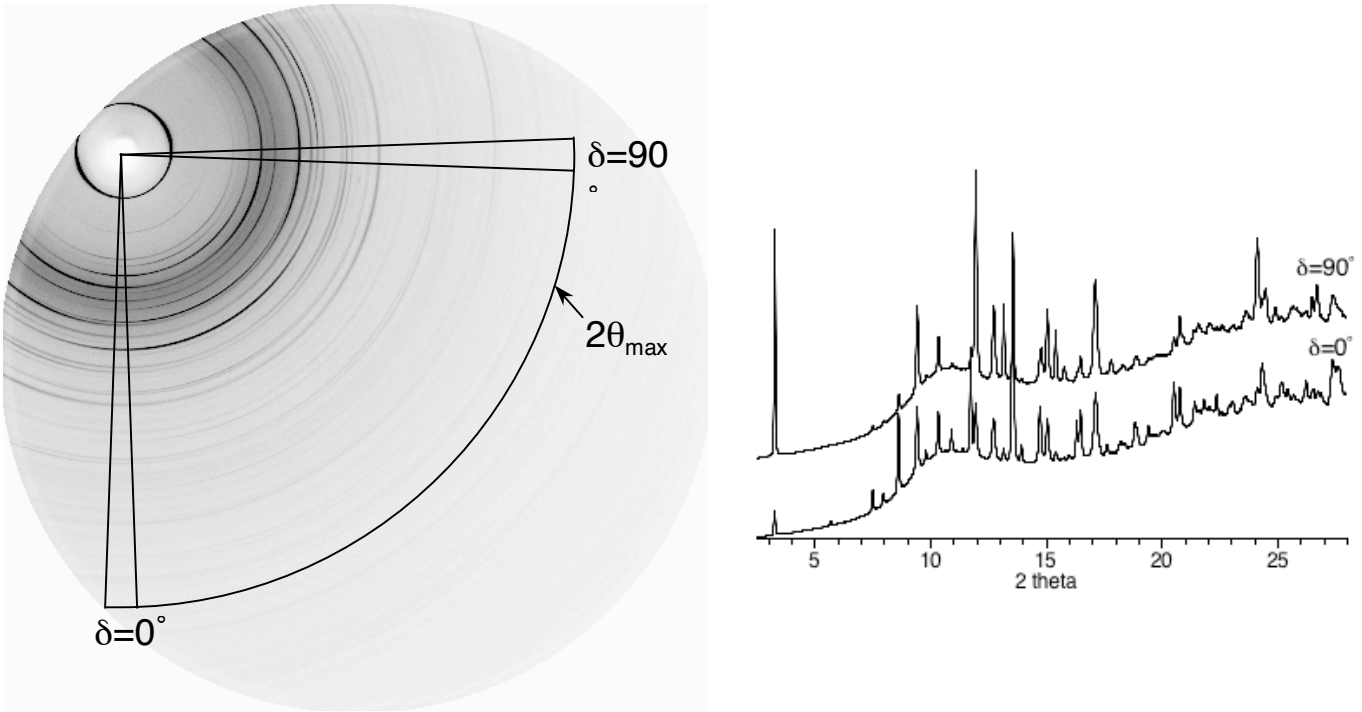

Figure 1. Imaging plate frame for E-401 at ψ=0° showing two of the 19 radial wedges used to generate the diffraction patterns for different tilt angles δ (left), and the corresponding diffraction patterns (right). Note the significant differences in peak intensities for the two different sample tilts.

Analysis of these data showed that the scaling between imaging plate frames is critical, and an alternative to the rather inaccurate monitor counts recorded on the MarResearch file was devised. Reasoning that the background should be independent of the sample rotation (assuming the sample is bathed in the beam), scaling was based on a set of selected background points. This proved to be a reliable procedure, and eliminated some anomalies in the pole figures (plots of intensity variation of a single reflection as a function of sample orientation) that had been noted in earlier analyses. Intensities of all reflections in all 1368 patterns were extracted, and the crystallite orientation distribution function was determined from these data. Then, taking into account the preferred orientation factor for each reflection at each orientation, a single set of reflection intensities was extracted from all 1296 unique diffraction patterns simultaneously. Attempts to solve the structure using these more single-crystal-like data are currently in progress.

References

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