

	Experiment title: Application of a new method of structure determination using transmission powder diffraction data to some textured polycrystalline samples	Experiment number: CH-1318
Beamline: BM01A	Date of experiment: from: 19-Jul-02 to: 22-Jul-02 7-Feb-03 9-Feb-03	Date of report: 27-Feb-03
Shifts: 9	Local contact(s): Silvia Capelli	<i>Received at ESRF:</i>
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Report:

These experiments are part of a larger project devoted to the development of a method of crystal structure determination using transmission powder diffraction data from textured samples. Full datasets were collected on one test sample (zeolite ZSM-5) and on several samples whose structures are unknown. Limited tests of the experimental setup were also performed.

The software for the data analysis (program *Expol*) was completed during this period. Before analysis of the data collected on the materials with unknown structure can begin, the viability of the software needs to be checked, so the data collected on ZSM-5 (a zeolite with a complex but known structure) was examined first.

Normally, a full dataset consists of two sets of 36 imaging plate frames (each corresponding to a 5° rotation of the textured sample). One set is collected with a short sample-detector distance (e.g. 240mm) to maximize the *d*-spacing range, and the other at a longer distance (e.g. 400mm) to get maximum resolution of peaks. Each frame is divided

into 72 radial wedges to yield a total of $2 \times 36 \times 72 = 5184$ powder diffraction patterns for data analysis.

Such data were collected on ZSM-5, and these have been analyzed. First, the reflection intensities were extracted from all patterns (ca 24h computing time per set of 36 frames, typical data shown in Figure 1). Then the texture (orientation distribution function) was determined using intensities extracted for single reflections (see Figure 2). Finally, a single set of single-crystal-like intensities was extracted from all data.

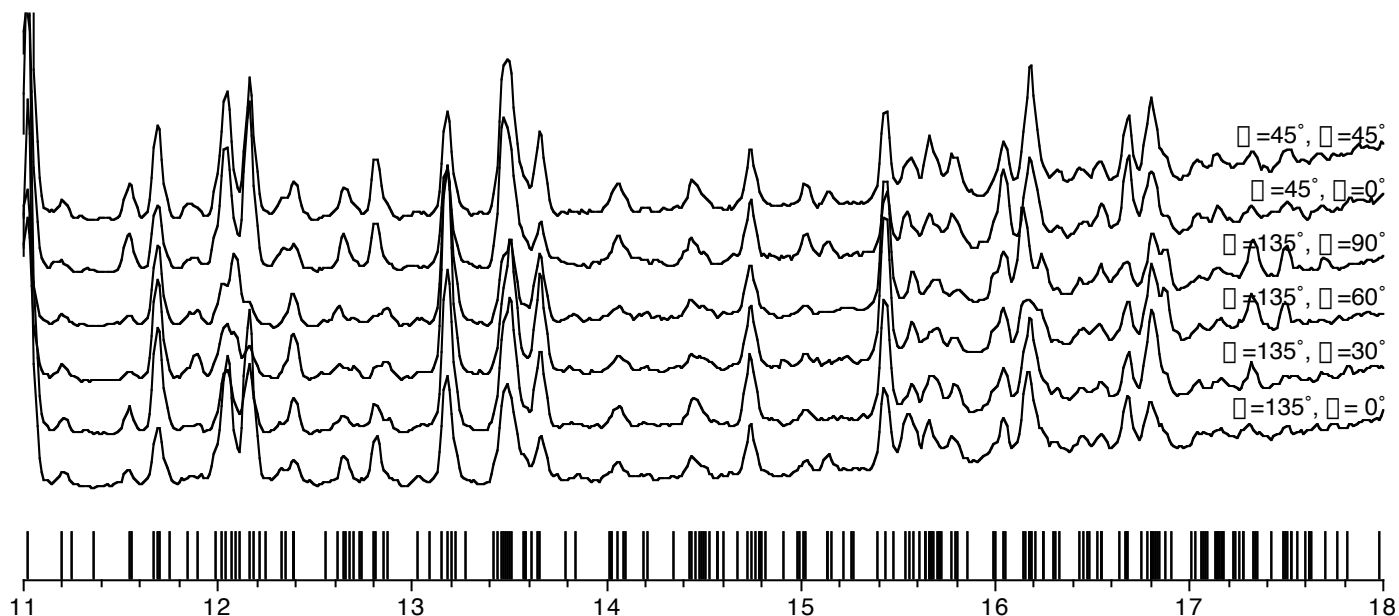


Figure 1. Sections of selected powder patterns collected for different orientations (ϕ : rotation, χ : tilt) of the textured ZSM-5 sample ($a = 0.70056 \text{ \AA}$). Note the significant intensity differences.

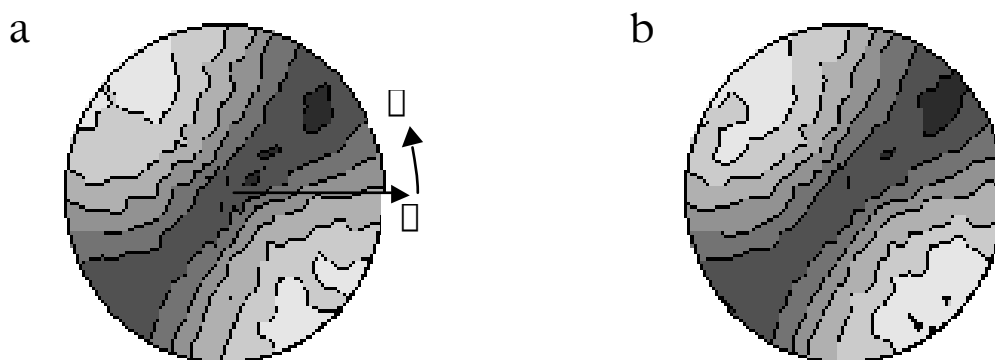


Figure 2. Typical pole figure (reflection 102, $5.99^\circ 2\theta$): (a) measured, and (b) calculated from the determined texture. Darker regions indicate the sample orientations yielding the higher intensity for this reflection.

Detailed comparison of these intensities with those calculated for the known structure, those extracted from a normal powder diffraction pattern, and those from a simulated dataset is now in progress.

Analysis of the other datasets collected during these experiments will be evaluated as soon as these software tests have been completed.