	Experiment title: Using a texture approach to determine the structures of complex polycrystalline materials	**Experiment number**: CH-898
Beamline: BM01B	**Date of experiment**: from: 26-Oct-2000 to: 3-Nov-00	**Date of report**: 29-Aug-01
Shifts: 21	**Local contact(s)**: Hermann Emerich	*Received at ESRF:*

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Report:

Textured samples of two materials with complex structures were prepared for this experiment. One was the aluminophosphate phase G (monoclinic, a = 16.682 Å, b = 26.129 Å, c = 9.869 Å, β = 90.4˚), and the second an organic pyrollidine ($C_{15}H_{22}N_2O_6S \cdot Na \cdot H_2O$, monoclinic, a = 11.107 Å, b = 6.249 Å, c = 29.778 Å, β = 103.5˚). The former (needle morphology) was prepared using a smear technique and a polystyrene/THF matrix, and the latter (platelet morphology) by sedimentation in water. Unfortunately, due to a vacuum problem on the beamline, only one sample (phase G) could be measured.

To determine how the crystallites were oriented in the sample, pole figure data on nine non-overlapping reflections were collected. These showed significant intensity differences as a function of sample orientation, so it was assumed that the sample was well textured. Then full diffraction patterns were collected at five different sample orientations that were expected to yield large intensity contrasts (Figure 1).

Subsequent analysis of the pole figure data, however, showed them to be internally inconsistent. Even when those pole figures with

poor statistics were removed from the calculation, no satisfactory description of the crystallite orientation distribution could be found. At this point, it was suspected that the sample was not homogeneous, so the different sample orientations, which are measured with different parts of the sample illuminated, could not be compared properly.

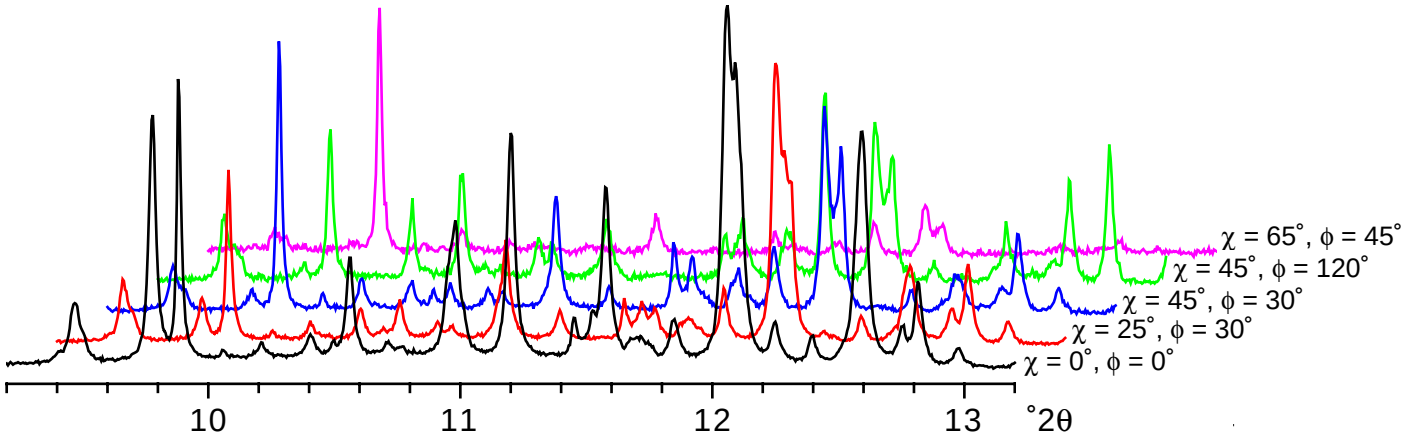

Figure 1. Portions of the diffraction patterns measured at different sample orientations, showing the changes in intensity as a function of sample orientation.

As part of the preparation for experiment MI-385, a portion of the sample used for this experiment was cut out and measured in transmission mode with an image plate detector in the laboratory. The origin of the problem then became apparent. There were some larger crystallites in the sample giving rise to large spots on the powder rings. With the reflection geometry used for this experiment, such problems only come to light during the data analysis. Even after careful preparation of a new sample, the synchrotron data showed that the spots had not been eliminated completely (Figure 2).

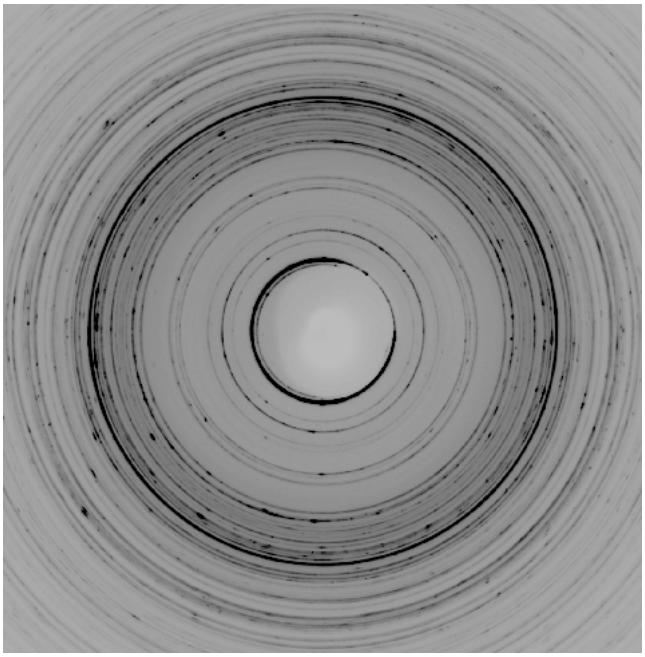

Figure 2. Synchrotron image plate data collected in transmission mode showing spots arising from the presence of larger crystallites within the sample.