| | **Experiment title:** Using a texture approach to determine the structures of complex polycrystalline materials | **Experiment number**: CH-815 |
|---|---|---|
| **Beamline**: BM01B | **Date of experiment**: from: 18-Feb-2000 to: 29-Feb-00 | **Date of report**: 20-Aug-01 *Received at ESRF:* |
| **Shifts:** 21 | **Local contact(s)**: Hermann Emerich | |

**Names and affiliations of applicants** (* indicates experimentalists):

***Thomas Wessels**

***Sinisa Prokic**

**Lynne McCusker**

**Christian Baerlocher**

**Laboratorium für Kristallographie, ETH, Zürich, Switzerland**

## Report:

Data on two materials with complex crystal structures were collected during this experiment. One was a triclinic ($a$=40.12 Å, $b$=10.29 Å, $c$=10.24 Å, a=90.7˚, b=91.7˚, b=81.9˚) zincosilicate related to VPI-7, and the other a monoclinic ($a$=17.2481 Å, $b$=4.7753 Å, $c$=10.5766 Å, b=98.80˚) organic compound (D-glycero-D-galacto-non-2-enonic acid). In both cases, the high-resolution powder diffraction pattern collected on a (randomly oriented) capillary sample proved to be insufficient for structure solution. It was hoped that a measurement of a textured sample, in which the crystallites had been oriented intentionally, might allow more of the overlapping reflections to be resolved.

Therefore, a textured sample of each was prepared by combining the polycrystalline material with a viscous solution of polystyrene in tetrahydrofuran and then smearing a thin film on a glass slide. When the film had dried, another layer was added. This process was continued until the specimen was thick enough for data collection (ca. 0.3mm).

To determine how the crystallites were oriented in the samples, pole figure data on eleven non-overlapping reflections were collected for the zincosilicate and on eight for the organic material. Then full diffraction patterns were collected at five different sample orientations for both samples.

In both cases, the pole figures show that the samples are textured (see Figure below). However, the different pole figures do not appear to be self consistent.

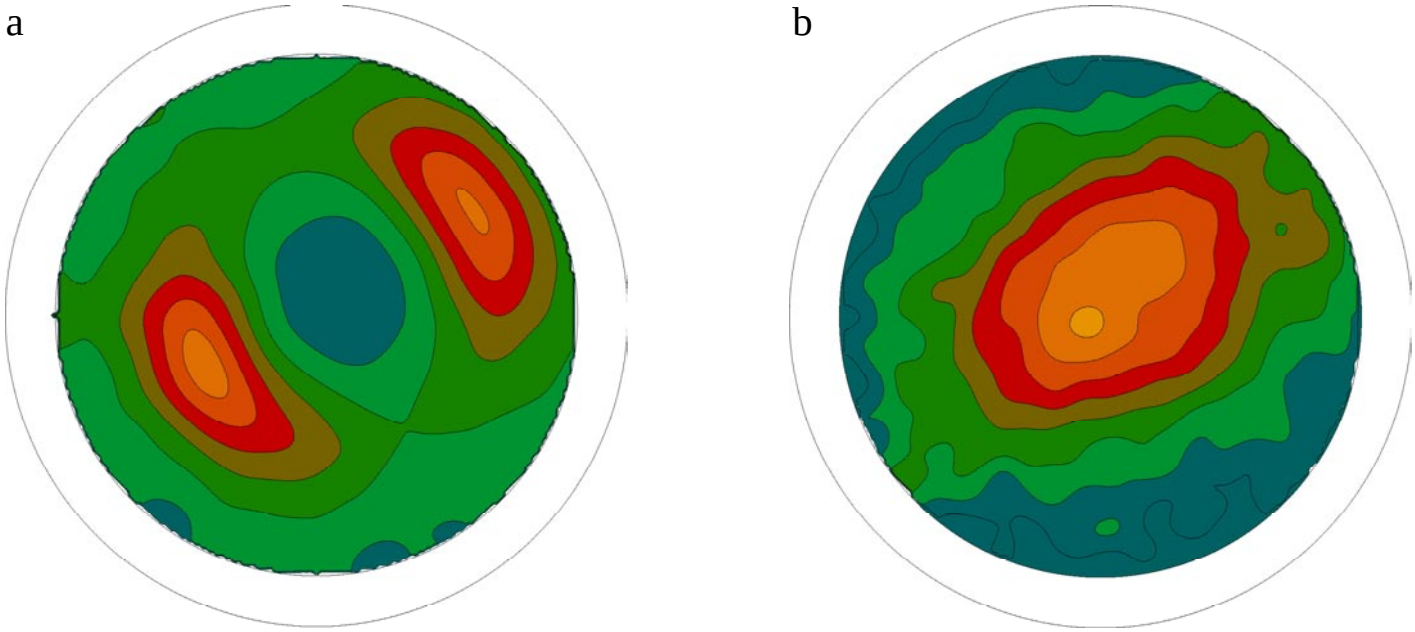


Figure 1. Pole figure for (a) the 310 reflection of the D-glycero-D-galacto-non-2-enonic acid sample, and (b) for the 056 reflection of the zincosilicate.

In the case of the organic material, the discrepancy is probably due to inhomogeneities in the texture of the sample, but for the zincosilicate it may be that the indexing is not yet quite correct. Although considerable effort has been put into the latter, so far no completely satisfactory solution has been found. The origin of the problem can be seen in the high-resolution pattern shown in Figure 2. The density of lines is so high that reliable assignment of triclinic indices is very difficult.

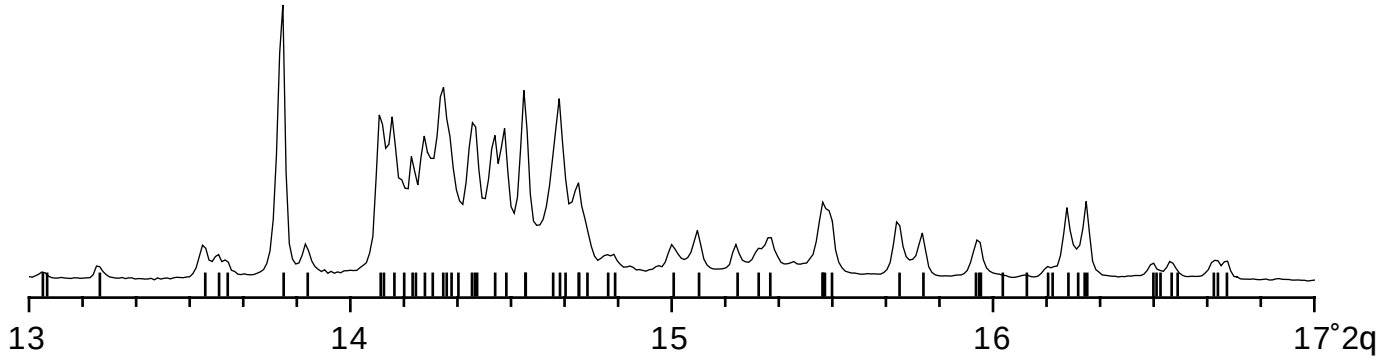


Figure 2. Small portion of the high-resolution diffraction pattern of the zincosilicate phase. Tick marks indicate reflection positions for just one of the possible indexing solutions.