



	Experiment title: Diffuse scattering of single crystal CaTiOSiO ₄ at high pressure	Experiment number: HS-788
Beamline: BM-01	Date of experiment: from: 09.06.1999 to: 14.06.1999	Date of report: 21.02.2000
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Report:

- (i) The main goal of the experiment HS-788 was to search for diffuse scattering of the mineral titanite (CaTiOSiO₄) at pressures under the phase transition P21/a <-> A2/a at 3.5 GPa (i) [2],[4]. Such diffuse scattering is known for the intermediate HT-phase of titanite as well as for natural specimens at ambient conditions. This diffuse scattering is indicative of a local order of the Ti-out-of-center distortion within domains. Its presence also in the HP A2/a phase would thus be an indication for the atomic mechanism (symmetrization vs. long range disorder) of this HP-phase transition. For this purpose, a single crystal titanite was measured at various pressures in the range between 0.001 to 3.7 GPa in a Diamond Anvil Cell (DAC). The phi rotation varied from 335⁰ to 55⁰ in 5 seconds with a Δphi =1⁰ per step.
- (ii) A second goal of this experiment was the first test of the ETH-Diamond Anvil Cell with respect to its use at the SNBL.

(i) Data processing was done with the program packages fit2d and KUMA. Problems occurred with fit2d in so far that the background could not be properly subtracted. This problem could however be solved using a self-learning algorithm (“Ewald explorer”) implemented in KUMA. Subsequent data processing of data taken from a natural specimen at ambient conditions for which diffuse scattering is expected revealed strong diffuse streaks (fig. 1) This demonstrates the principle possibility of detecting diffuse scattering even through the Compton background of a diamond anvil cell.

Using the program KUMA, orientation matrices for titanite at 2.6 GPa and 3.7 GPa could easily be found, which demonstrates the feasibility of the MAR-system for small-molecule single crystal diffraction in a diamond anvil cell (small data redundancy, additional diffraction peaks from rubies and diamonds). The unit cell refined at these pressures were in accordance with data from the literature [1], [3] (2.6 GPa: $a = 6.989(10)$ Å, $b = 8.616(10)$ Å, $c = 6.492(9)$ Å, $\beta = 113.32(14)^\circ$; for 3.7 GPa: $a = 6.956(9)$ Å, $b = 8.587(9)$ Å, $c = 6.457(8)$ Å, $\beta = 113.25(13)^\circ$)

In natural titanite the impurities seem to sustain these scattering also after passing the phase transition pressure of 3.5 GPa (fig.2). This reaffirms and extends the results of [2] and [4], and validates the above-mentioned assumption of long range disorder in natural titanite.

Ongoing data analysis focuses on the extraction and interpretation of both the Bragg intensities as well as the diffuse scattering, which is expected to contribute to the understanding of the nature of exact mechanism of the HP-phase transition in titanite

(ii) The Diamond Anvil Cell used in the experiment proved to be perfectly suitable for the SNBL set-up. It was especially encouraging to see that with our modified diamond anvil cell, we can easily work at wavelengths between 0.7 and 0.8 Ångstroms. This bears further advantages as to the filtering of gasket signals by the gasket itself.

- [1] Angel, R.J., Kunz, M., Miletich, R., Woodland, A.B., Koch, M. and Xirouchakis, D. (1999), Phase Transitions, 68, 553-543
- [2] Higgins, J.B. and Ribbe, P.H. (1976), American Mineralogist, 61, 878-888
- [3] Kunz, M., Xirouchakis, D., Lindley, D.H. and Häusermann, D. (1996), American Mineralogist, 81, 1527-1530