EUROPEAN SYNCHROTRON RADIATION FACILITY
INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

# Experiment Report Form

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|---|---|---|
| | **Experiment title:** Development of micro-diffraction techniques for characterizing complex materials | **Experiment number**: 01-02-808 |
| **Beamline**: BM1 | **Date of experiment**: from: 26/07/2008 to: 30/07/2008 | **Date of report**: 25/08/2008 |
| **Shifts:** 12 | **Local contact(s)**: Dr. Philip Pattison | *Received at ESRF:* |

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

Dr. Rainer DAEHN*

Dr. Erich WIELAND*

Paul Scherrer Institut, Laboratory for Waste Management,

CH-5232, Villigen PSI.

## Report:

Cement-based materials are complex heterogeneous mixtures with discrete micro-scale particles. Due to this in spite of frequent use of cements for immobilization and solidification of toxic and radioactive waste around the world, the atomic/molecular level understanding of chemical processes in cement is still poorly understood. The aim of the proposed study was to collect XRD patterns on selected areas of the cement thin sections for the characterization of the mineral composition in the complex cement matrix. The use of classical powder diffraction in combination with Rietveld refinement on cementitious systems is well established (e.g., [1]). **To the best of our knowledge, the use of synchrotron-based XRD with a high spatial resolution to characterize the complex mineral assemblage in cements was novel.**

X-ray diffraction measurements were performed on thin sections of a sulphate-resisting Portland-cement using Mar345 system available at BM01. It was shown that crystalline phases can be identified in selected regions of the samples. Due to the restricted beam spot size, which was about 100 μm, it was possible to keep a limited number of crystals in the beam during data collection (Fig. 1) and therefore, typical diffraction pattern contains only spots from selected crystallites (Fig. 2.). Their indexation with CrysAlis program allowed crystals of Portlandite ($Ca(OH)_2$) and Alite ($Ca_3SiO_5$) to be clearly identified. Structures of

both minerals could be refined using the anisotropic approach of thermal parameters against collected in situ intensity data typically having 0.9 Å highest resolution (Table 1).

The results obtained will be used as a basis for further X-ray diffraction studies of cements. Publication based on the data is now beeing prepared.

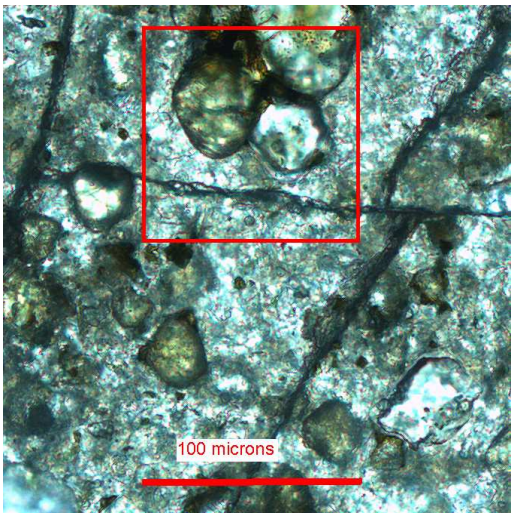


Fig. 1: Optical image of cement thin section in transmitted light showing studied area (red rectangle).

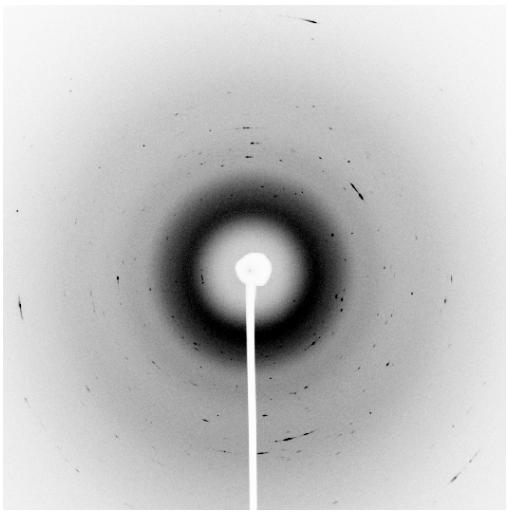

Fig. 2: Micro-diffraction images of a cement sample showing diffraction spots.

Table 1. Typical parameters of X-ray experiments and structure refinement.

| Parameter | Portlandite (I) | Portlandite (II) | Alite |
|---|---|---|---|
| Highest resolution | 0.90 | 0.90 | 0.90 |
| Space group | $P\bar{3}m1$ | $P\bar{3}m1$ | R3m |
| Unit cell, Å | *a*=3.6, *c*=4.9 | *a*=3.6, *c*=4.9 | *a*=7.06, *c*=24.97 |
| Completeness, % | 86(91)[1] | 81(90)[1] | 88(94)[1] |
| Redundancy | 1.6(1.5)[1] | 0.8(0.9)[1] | 2.4(2.6) |
| Rint[2] | 0.018(0.066)[1] | - | 0.055(0.049) |
| I/σ | 102(30)[1] | 77(39)[1] | 35(28) |
| R1[3] | 0.116 | 0.053 | 0.128 |

[1] values in parentheses correspond to the outer resolution shell 1.0-0.9 Å.
[2] Rint = Σ | Fo - Fo(mean) | / Σ [ Fo]
[3] R1 = Σ | |Fo| – |Fc| | / Σ |Fo|

## References.

1. Scrivener, K.L., et al., *Quantitative study of Portland cement hydration by X-ray diffraction/Rietveld analysis and independent methods.* Cement and Concrete Research, 2004. 34: 1541-1547.