

# Preliminary Experiments with High-Energy X-Ray Diffraction as Alternative Method for *in-situ* Studies

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The synthesis of new compounds in the field of solid-state chemistry is often the result of intensive explorative work by varying the large number of different reaction parameters in a systematic way. This trial-and-error approach is still the common way to prepare new materials following the solvothermal route. Despite of the large number of reports dealing with the solvothermal synthesis of new compounds the number of studies of the crystallization mechanisms occurring under solvothermal conditions is still low and the solvothermal method a kind of black-box chemistry. In the last decade, the number of publications presenting results of solvothermal synthesis studies using *in-situ* energy dispersive X-ray diffraction (EDXRD) has grown rapidly.

The great advantages of *in-situ* EDXRD experiments have now been well established, nevertheless a "white beam" of X-rays from the synchrotron is crucial to perform the investigations. Unfortunately, due to the limited resource of the white beam suppliers and high demand from researchers, the number of reports is still restricted. The energy of such white beams allows the application on thick samples or even the penetration of incident beams into different reaction vessels/materials to monitor chemical syntheses under real reaction conditions. During the last few years we studied several reactions under solvothermal conditions with *in-situ* EDXRD [1-9] at the beamline F3, HasyLab, Desy.

As the beamline F3 will stop functioning in 2012, we began to search for alternatives. The accessible energies range from 60 to 150 keV provide at the high-energy X-ray scattering diffractometer on beamline BW5 satisfies our experimental requirements mentioned above.

In 2010, we had the opportunity to carry out some test experiments at BW5. Initially, reactant chemicals and product samples were measured in glass capillaries to estimate the angle range and time resolution. Afterwards the different reaction vessels of our *in-situ* cell (e.g. aluminium autoclaves with Teflon liners and glass tubes) were irradiated to see their influence/effect in the diffraction pattern and to run stability tests. Finally, a few reference suspensions were measured in the sample environment described in Figure 1 caption.

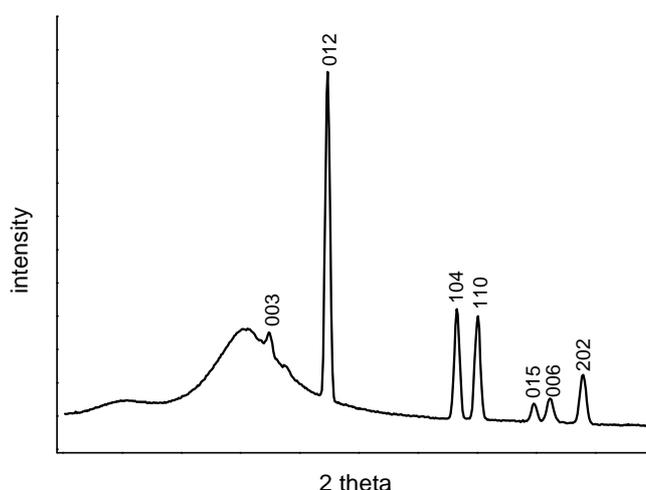


Figure 1: Diffraction pattern of a Sb suspension with labelled Bragg peaks, measured in our *in-situ* cell (aluminium autoclave with glass vessel) at BW5 ( $E = 100.3211$  keV).

The high-energy X-ray scattering diffractometer at BW5 could be used for *in-situ* studies of solvothermal reaction, if we find a suitable way for the further evaluation of the data.

## References

- [1] L. Engelke, M. Schaefer, M. Schur, W. Bensch, *Chem. Mater.* **13**, 1383 (2001).
- [2] L. Engelke, M. Schaefer, F. Porsch, W. Bensch, *Eur. J. Inorg. Chem.* 506 (2003).
- [3] R. Kiebach, M. Schaefer, F. Porsch, W. Bensch, *Z. Anorg. Allg. Chem.* **631**, 369 (2005).
- [4] A. Michailovski, J.-D. Grunwaldt, A. Baiker, R. Kiebach, W. Bensch, G. R. Patzke, *Angew. Chem.* **117**, 5787 (2005).
- [5] R. Kiebach, N. Pienack, M.-E. Ordolff, F. Studt, W. Bensch, *Chem. Mater.* **18**, 1196 (2006).
- [6] A. Michailovski, R. Kiebach, W. Bensch, J.-D. Grunwaldt, A. Baiker, S. Komarneni, G. R. Patzke, *Chem. Mater.* **19**, 185 (2007).
- [7] R. Kiebach, N. Pienack, W. Bensch, J.-D. Grunwaldt, A. Michailovski, A. Baiker, T. Fox, Y. Zhou, G. R. Patzke, *Chem. Mater.* **20**, 3022 (2008).
- [8] N. Pienack, C. Näther, W. Bensch, *Eur. J. Inorg. Chem.* 937 (2009).
- [9] Y. Zhou, E. Antonova, W. Bensch, G. R. Patzke, *Nanoscale* **2**, 2412 (2010).