European Mineralogical Conference
Vol. 1, EMC2012-339, 2012
European Mineralogical Conference 2012

In-Situ X-Ray Micro-Diffraction Analysis of Reaction Products in Heterogeneous Concrete Samples

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Synchrotron-based X-ray micro-diffraction techniques with a beam size focussed down to a few micrometres or less allow a variety of novel applications in the field of diffraction experiments on fine-structured materials with a high spatial resolution. In addition to the state-of-the-art investigation of pure crystalline materials that are only available at very small sizes and very low quantity, micro-diffraction experiments also allow local probing of heterogeneously composed materials that show chemical and structural zonation on a (sub-)micrometre scale, $e.g.$ components in nanotechnology and samples of interest in environmental sciences, geochemistry and engineering, such as structural alterations or reaction products forming at phase contacts.

In the present study, reaction products formed during the alkali-silica-reaction (ASR) in concrete structures have been investigated. The ASR is an important deterioration process in concrete, leading worldwide to severe damages in buildings, dams, bridges *etc.*. Reaction products formed due to interaction of amorphous or crystalline SiO_2 in aggregates with the alkaline pore solution of concrete produces expansive pressure and, as a consequence, gives rise to damage of concrete structures. Knowledge of the mechanics leading to this expansion during the course of ASR is still limited. Analysis and identification of the reaction products formed on a micrometre scale is the key to further understanding ASR.

Samples taken from an ASR damaged concrete structure showing veins of ASR reaction products were prepared as thin sections with a thickness of about 20 μm. Expansion cracks of aggregates due to ASR in the available samples have typically a cross-section in the range of a few tens of micrometres. Synchrotron-based X-ray micro-diffraction data were collected *in-situ* along these veins, $i.e.$ ASR products were probed within their original environment. Powder diffraction data analysis and analysis of single crystal diffraction patterns obtained from micrometre or sub-micrometre sized, micro-crystalline grains in the ASR zones were carried out. However, due to inevitable multi-phase sampling even with a sub-micrometre beam and the intricate handling and positioning of the samples under the beam, the quality of the diffraction data is limited, thus complicating phase identification and refinement. A structural model of the reaction product will be presented based on the current status of data analysis.