QUANTITATIVE IN-SITU X-RAY DIFFRACTION ANALYSIS OF EARLY HYDRATION OF PORTLAND CEMENT AT DEFINED TEMPERATURES

C. Hesse[(1)], F. Goetz-Neunhoeffer[(1)], J. Neubauer[(1)], M. Braeu[(2)], P. Gaeberlein[(2)], M. Degenkolb[(3)]
[(1)] Mineralogy, University of Erlangen-Nuremberg, D-91054 Erlangen, Germany
[(2)] BASF Construction Chemicals GmbH, D-83308 Trostberg, Germany
[(3)] PCI GmbH, D-86159 Augsburg, Germany

X-ray diffraction (XRD) is widely used for the determination of phase composition of Portland cement powders. To understand interactions between additives and the cement phases also a time resolved quantitative phase analysis of the early hydration at defined temperatures is necessary.

Technical white cement was mixed with water and prepared in a custom-made sample holder maintaining the surrounding temperature at a constant value. The sample was covered by Kapton film which prevents water evaporation. A D5000 diffractometer (Bruker) equipped with a SolX energy dispersive detector was used. 88 diffraction patterns were recorded within 22 hours of hydration.

Each diffraction pattern was refined by the Rietveld method with the software Topas 3.0 (Bruker). In consequence of the used Kapton film, a background model must be developed for the diffuse diffraction maximum of the material. The lattice parameters, crystallite size, and microstrain were refined for the hydrate phases. The parameters for the clinker phases, calcite, and anhydrite were determined by measurements of dry cement and were fixed during refinement of the cement paste. The content of X-ray amorphous C-S-H gel in the paste linked with the formation of portlandite was calculated from the content of portlandite. The quantification shows the progress of the silicate and the aluminate reaction and the dependence of their kinetics on the temperature. The link between the formation of ettringite and the dissolution of the aluminate phase and anhydrite could be shown.

The measurements and their Rietveld refinements exhibit a very good reproducibility. Additionally, the noise of the phase content development within 22 hours is very low. The investigations have shown that quantitative in-situ XRD of cement hydration can be performed also with lab diffractometers.