A NEW SAMPLE HOLDER FOR FAST XRD INVESTIGATION ON UHPC

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Introduction

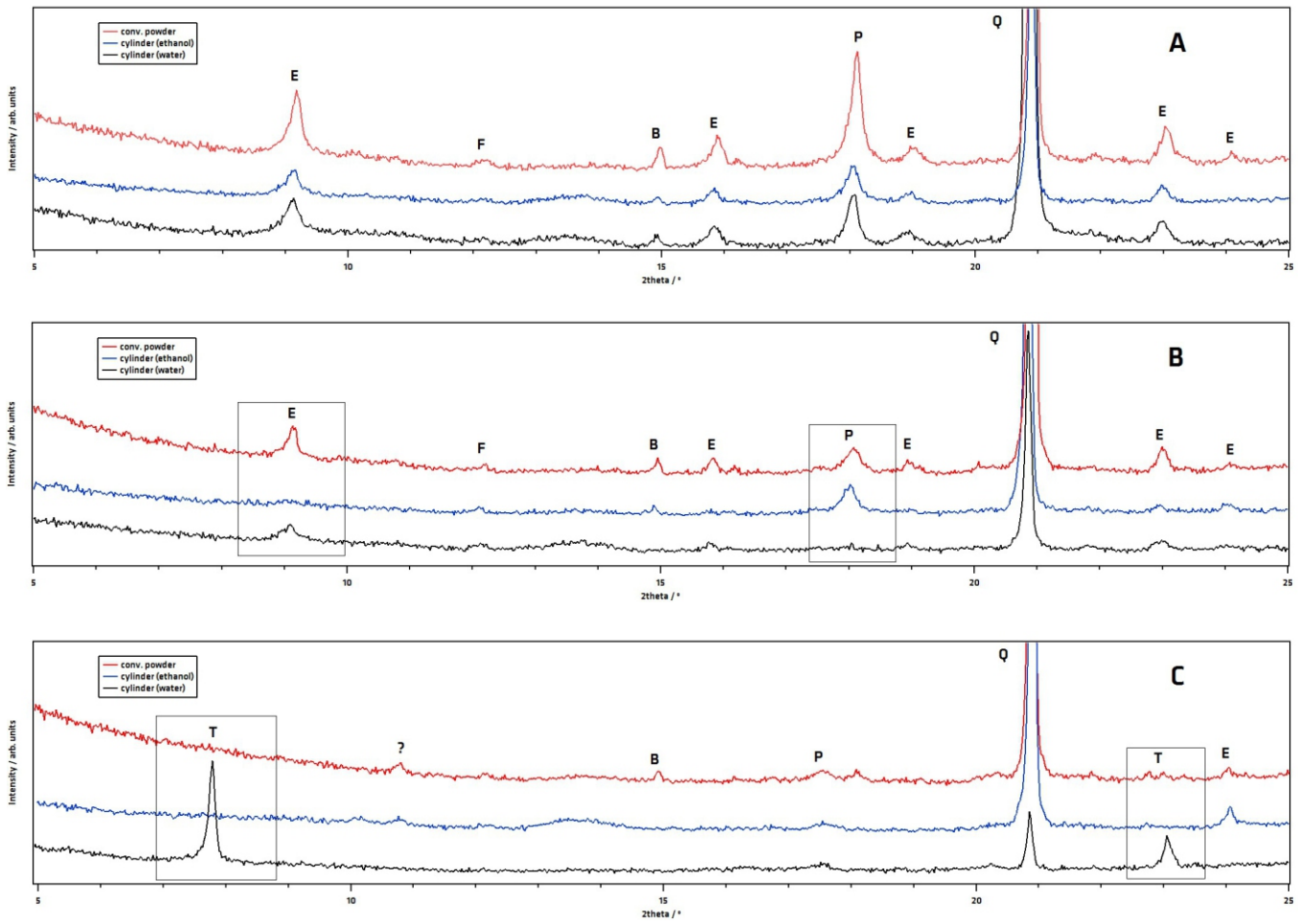
One of the most challenging tasks for powder x-ray diffraction analyses (XRD) is the reliably preparation of samples, in particular when a comprehensive series of experiments has to be investigated. The quality of the collected data strongly depends to the quality of the preparation and deposition [1,2,3]. In concrete science, one has to deal with comprehensive series of experiments, where superficial phase identification is needed. If the particle size and the statistical distribution of the crystals is guaranteed, time could be reduced by measuring the solid sample. It has been commonly accepted that a particle size of <325 mesh (44µm) is satisfactory for qualitative x-ray phase analyses [1]. UHPC has an average particle size < 50 µm and no preferred orientation of the crystals. Aim of this study is to enable in situ measurement of small UHPC cylinders with slight preparation, employing a new XRD sample holder.

Experiments

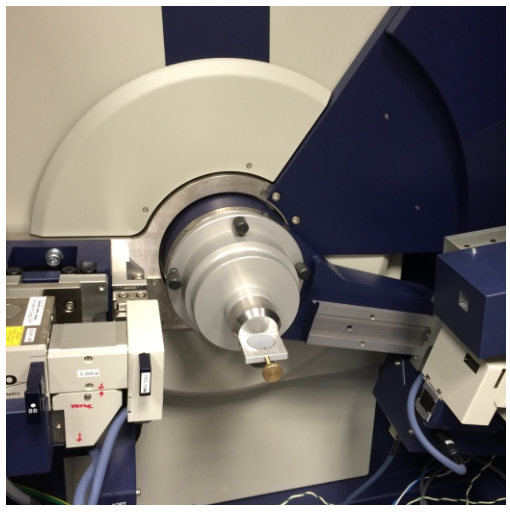
Small UHPC cylinders were prepared and stored for 28 d in water to achieve the final strength. Afterwards the cylinders were retreated with a hot water bath (95°), respectively with autoclave (185° 1.1 MPa) to increase the mechanical strength (for more details see [4, 5]). To get the required flat surface cylinders where grinded with water respectively ethanol as lubricant. Additionally, samples were conventionally prepared as powder.

Results and Discussion

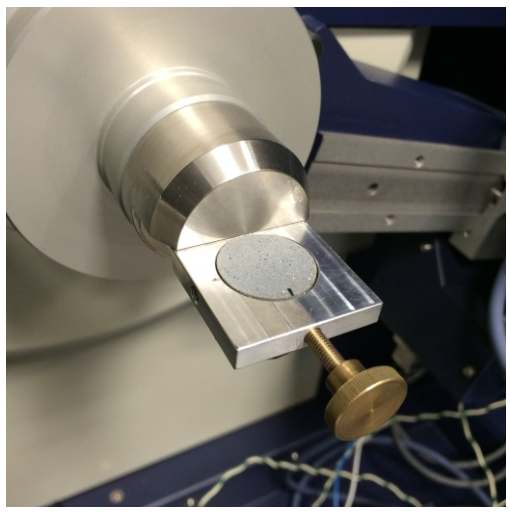
The measurement of solid UHPC cylinders show a good signal to noise ratio. The 28 days water storage samples exhibit negligible differences in intensity between the powder and the ethanol, respectively water grinding. The hot water storage samples reveal stronger differences, the ethanol grinded samples lacks of ettringite, while portlandite is absent in the water grinded sample. The water grinded sample of the autoclave series shows a strong reflection of tobermorite at the surface which is missing at the bulk powder sample. In summary, solid sample analyse of fine-grained concrete enable a fast and reliable phase analyse with the suitable lubricant. Furthermore, depth resolved measurements are possible with additional benefits towards conventional powder diffraction analyses.



Diffractogram of (A) 28 d water storage, (B) hot water storage, (C) autoclave
The identified phases ettringite (E), brownmillerite (F), portlandite (P), belite (B), quartz (Q) and tobermorite (T) are marked with the corresponding letter. Regions of interest are marked with a black box.



Bragg-Brentano Diffractometer
Samples were measuerd with the Rigaku Ultima 4 from 5- 25 ° 2Theta for 0.5 min/° with 0.02° stepwith.



New Sample Holder
New developed cylinder sample holder for x-ray diffraction analyses of UHPC cylinders.

UHPC Composition

comp.	wt%
cement	34.7
microsilica	5.6
quartz powder	8.6
quartz sand	40.7
superplasticizer	1.7
water	8.7
sum	100

Appendix
Constructional drawing for varios sample geometries in DXF-format will be provided for free, just send me an email to sebastian.simon@bam.de.

Reference

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