Durability of Ultra-High-Performance Concrete

by C. Pfeifer, B. Möeser, C. Giebson, and J. Stark

Synopsis: In recent years ultra-high-performance concrete (UHPC) has gained more interest in the concrete construction industry due to the expected high durability of UHPC, as well as extended architectural opportunities. Compared to normal concrete it is possible to build filigree and lighter structures with ultra-high-performance concrete.

The aim of this study was to evaluate the durability of different UHPC mixtures regarding alkali-silica reaction (ASR) and delayed-ettringite formation (DEF). UHPC prisms were exposed to different temperature and moisture conditions in a special climate simulation chamber. Scanning electron microscopy (SEM) was used to determine possible deterioration of UHPCs. Results of microscopic investigations show that products of ASR are only locally enriched. An ettringite growth was observed on microcracks (< 10 μm) in intentional pre-damaged samples or close to incompletely hydrated clinker grains. Macroscopic deteriorations due to ASR or ettringite growth could not be detected. However, steel fibers of UHPC were affected by corrosion.

Keywords: delayed ettringite formation; durability; ultra-high-performance concrete.

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INTRODUCTION

Structures made of ultra-high-performance concrete (UHPC) are getting more attention in the concrete construction industry. Due to the extremely dense microstructure of UHPC a high resistance against different kinds of aggressive media is assumed. In the last 2 years, several studies have shown results on chloride penetration depth, carbonation, resistance against sulfate, and chemical attack or freezing and thawing.[1-8] So far, no test results on the durability of UHPC structures under European climate conditions have been published.

Examinations by the authors with electron microscopy on polished sections of UHPC (Fig. 1) displayed a high amount of incompletely reacted clinker particles in the UHPC microstructure compared to standard mortar (Fig. 2). These unhydrated particles could present a potential for damage if water or aqueous solutions would penetrate into the concrete microstructure.

Results of the present study are part of a long-term study on durability of UHPC. UHPC prisms were exposed to alternating climate conditions by means of the cyclic climate storage, using a test method developed at the F. A. Finger-Institute for Building Materials Science (FIB). Of special focus is the impact of microcracks and the penetration of fluids on the durability of UHPC. The cyclic climate storage simulates the climate conditions in Central Europe (drying, moistening, freezing and thawing) in an accelerated manner on constructions exposed to weathering. The test is especially suitable for problems related to ASR, and is described by Seyfarth and Stark.[9,10] The cement and silica fume (SF) content in UHPC mixtures is generally high. Normally UHPC mixtures contain between 600 and 1000 kg/m^3 (1010 and 1685 lb/yd^3)of cement,[11] whereas normal concretes contain approximately 400 kg/m^3 (675 lb/yd^3) of cement. UHPC generally contains silica fume contents between 10-15 wt.-% based on cement.[12] During UHPC production silica fume is added as a dry component. Thus, homogeneous SF distribution is difficult to obtain. For those reasons, it is necessary to investigate the potential durability of UHPC regarding ASR. Furthermore, the durability of UHPC has not been sufficiently investigated regarding DEF and corrosion of steel fibers.

MATERIALS AND METHODS

Materials and experimental setting

The investigations were performed on two UHPC mixtures, a fine grained UHPC mixture (M2Q-1) and a coarse-grained UHPC mixture (B4Q).[2] A normal portland cement (CEM I 52.5 R HS/NA) according to the German version of European DIN EN 197-1[13a]/ DIN EN 197-1/A-3[13b] and the following additives and aggregates were mixed: silica fume, two quartz powders with different grain sizes, quartz sand [0.125/0.5 mm (0.005/0.02 in.)], crushed basalt [0.25 mm (0.010 in.)], and superplasticizers based on polycarboxylate ether. Two different superplasticizers (Table 1) were used to achieve a sufficient workability. The mixture proportions, chemical and mineralogical composition, and the properties of the raw materials are shown in Tables 1 thru 4. In order to improve the ductility of ultra-high performance concrete, steel fibers [length/diameter: 9.0/0.15 mm (0.35/0.006 in.)] were used.

Ultra-high-performance concrete was mixed according to recommendations in Reference 2 with a compulsory mixer. Fig. 3 summarizes the sample treatment until the cyclic climate storage started. The concrete prisms [100 x 100 x 400 mm (4 x 4 x 16 in.)] were kept in molds at room temperature [20 °C (68 °F)] for 48 hours. After de-molding, two different subsequent treatments were performed. Samples of series WS were stored in water for 6 days, while samples of series HT were heat-treated in a regime at 90 °C (194 °F) maximum temperature for 48 hours, analogous to the heat treatment regime of Reference 2. Afterwards, prisms were stored for 6 days under standard conditions (20 °C [68 °F], 65 %RH). So far, there is no restricted heat treatment for UHPC in the German guidelines. According to the German guideline for heat treatment of concretes, issued by the German Committee for Reinforced Concrete (Deutscher Ausschuss für Stahlbeton (DAfStB)),[23] a heat treatment at 60 °C (140 °F) maximum is recommended for concrete category WF (moist environment) for normal concrete,[14] heavy-weight concrete.[14] and light-weight concrete.[15] At age of 8 days, the cyclic climate storage was started. On the 12th day after mixing, intentional pre-damage was done by a defined mechanical stresses. This was done to increase the permeability of the concrete and to accelerate possible damaging processes.[16] The procedure of mechanical pre-damage was reviewed with resonance frequency measurements.

Cyclic climate storage

Since 2001, an alternating climate test method (cyclic climate storage) has been used at the Finger-Institute for accelerated simulation of Central European climatic conditions, to assess the durability of specific concretes in outdoor structures.[9,10] Weather conditions in Central Europe—including drying, moistening, freezing and thawing—are simulated in an accelerated manner on structures exposed to weathering.

In a special walk-in climate simulation chamber (Feutron Klimasimulation GmbH, Type 3705/04) the concrete prisms were stored under defined cyclic alternating temperature and moisture conditions. One cycle (Fig. 4) lasted 21 days and consisted of 4 days drying at 60 °C (140 °F) (< 10 % RH), 14 days fog at 45 °C (113 °F) (100 % RH), and 3 days of freezing and thawing between +20 °C (68 °F) and –20 °C (–4 °F), according to CDF-Test. Test method standards can be taken from Reference 10. Investigations have shown that the amount of freezable water in UHPC is highly reduced.[7] No moisture uptake according to the frost pump takes place during frost exposure.

At the beginning of every cycle, mass and expansion of the prisms were measured. To avoid temperature influences caused by thermal expansion, the axial expansion of concrete prisms [100 x 100 x 400 mm (4 x 4 x 16 in.)] was always measured at 20 °C (68 °F). Deterioration of the concrete can be expected when expansion exceeds 0.4 mm/m (0.0004 in./in.).[10] This threshold value is only valid for unreinforced concrete. So far there have been no experiments with reinforced concretes. The cyclic climate storage is primarily used as the ASR-performance-test for concrete pavements.[9,10,17] In this case, 8 cycles (6 months) are usually sufficient to assess the potential for a deleterious ASR attack for European pavements with an intended service life of 20-30 years. In this study, the durability of UHPC was tested with the cyclic climate storage over 36 cycles (2 years).

Electron microscopy

The microstructure of UHPC was studied after every second cycle (42 days) and at final evaluation after the end of the cyclic climate storage (2 years). Various high resolution scanning electron microscopy (HR-SEM) imaging techniques were applied to determine deterioration or phase changes. Freshly fractured sample surfaces were examined by means of environmental scanning electron microscope (ESEM) in a water vapor atmosphere near 90% RH (WET-mode). The special features of this method are described elsewhere.[18,19] Polished sections of the UHPC microstructure were studied with high resolution backscattered electron (BSE) imaging techniques using conventional SEM or ESEM. The BSE image contrast is generated by varying average atomic number of the phases. This results in varying degrees of brightnesses in the image.

RESULTS

Results of expansion measurements for the two UHPC mixtures M2Q-1 (Fig. 5) and B4Q (Fig. 6) show that after 2 years of cyclic climate storage the highest expansion of 0.38 mm/m was measured for intentional pre-damaged prisms of mixture B4Q. For comparison purposes a normal concrete[20] [CEM I 32.5 R (Na_2O eq = 1.1 %), 400 kg/m^3 (25 lb/ft^3); 70% greywacke, 30% quartz sand] with W/C of 0.45 has been integrated in Fig. 5 through 6.

Microscopic examinations with different imaging techniques on fractured surfaces and polished sections show a delayed ettringite formation in cracks (width ≤ 10 μm) (Fig. 7) and near aluminate-rich normal portland cement clinker phases (Fig. 8). ASR was observed for the fine-grained UHPC mixture (Fig. 9), irrespective of the storage conditions (water storage, heat treatment, or cyclic climate storage). After several cycles of cyclic climate storage, steel corrosion was detected at the prism surface. At the end of the cyclic climate storage, steel corrosion and deterioration of the surrounding UHPC matrix were proven by SE imaging and electron probe microanalysis (EDX analysis) (Fig. 11-13).

DISCUSSION

Results of the present study show that the durability of two UHPC mixtures (M2Q-1, B4Q) is based upon three types of damage: corrosion of steel fibers, ASR, and DEF. Fig. 5 documents a minor expansion during the first two cycles of cyclic climate storage. It is assumed that this is a hygric expansion of the samples. Even after 2 years of cyclic climate storage, the expansion of the UHPC mixtures is low [< 0.4 mm/m (0.0004 in./in.)], regardless of water storage, heat treatment, or intentional pre-damage. For comparison reasons the expansion of a normal concrete[20] with an expansion >0.4 mm/m (0.0004 in./in.) and developed ASR is displayed in Fig. 5-6.

A closer evaluation of mixture M2Q-1 (Fig. 5) shows that the durability of UHPC is apparently influenced by the exposure conditions before the cyclic climate storage. From the beginning of the measurements, samples stored in water (WS) showed a lower expansion compared to samples with heat treatment (HT). The maximum expansion of prisms with WS is 0.14 mm/m (0.14×10^{-3} in./in.). The expansion of the heat-treated samples is significantly higher with a max. value of 0.25 mm/m (0.25×10^{-3} in./in.). Furthermore, it can be seen in Figure 5 that expansion of samples (series HT) is 50-60% higher than expansion of undamaged samples of series WS. The mixture B4Q (Fig. 6) showed slightly negative expansions for the undamaged series [between 0.0 and -0.07 mm/m (-0.07×10^{-3} in./in.)] irrespective of the storage conditions which are caused by shrinkage. Intentionally pre-damaged prisms with heat treatment exhibit the highest expansion with a value of 0.38 mm/m (0.38×10^{-3} in./in.).

Different imaging techniques of electron microscopy were applied to determine deterioration or phase changes in microstructure after cyclic climate storage. Due to the mix composition and to its optimized grading, UHPC has an extremely dense microstructure and seems to have high durability against chemical and physical attack. Comparison of SEM-BSE imaging of polished UHPC sections (hydration age 28 days) (Fig. 1) and a standard mortar (Fig. 2) reveals an extremely dense microstructure in UHPC. Furthermore, a high amount of unreacted clinker particles is also obvious. Due to a very low W/C (0.22-0.27) the amount of dissolved and reacted clinker particles in UHPC is considerably lower than in normal mortar/concrete (W/C = 0.4-0.5). Further investigations on polished sections of heat treated samples and results of powder diffraction with quantitative analysis[22] show that between an age of 5 days (immediate after heat treatment) and 28 days the amount of unreacted clinker particles does not decrease. After heat treatment no further hydration takes place due to a lack of water. During heat treatment the entire water is consumed, the hydration process is not proceeding. These results are in accordance with compressive strength results (not displayed). Between 7 and 28 days no more increase in compressive strength was observed. Polished sections of water stored samples show a decrease in unreacted clinker particles as hydration process proceeds. These high amounts of unhydrated clinker particles represent major damage potential if microcracks allow fluids to penetrate the concrete.

By means of electron microscopy a DEF was observed for the first time after the 4th cycle of climate storage in heat-treated and pre-damaged samples of mixture M2Q-1. Ettringite grows in cracks having a width smaller 10 µm.[21] Cracks which exceeded a width of 10 µm show a strong carbonation effect. Furthermore, DEF was observed after 8 cycles of climate storage close to aluminate-rich clinker grains in heat-treated undamaged samples (Fig. 8) and samples with water storage and intentional pre-damage.

Through insufficient dispersion of SF, locally isolated silica fume accumulations are present in the UHPC microstructure. ASR (Fig. 9 and 10) can take place irrespective of the exposure conditions. The typical ASR crack formation occurs isolated around the silica fume accumulations. Due to a lack of water (W/C = 0.22) the ASR-gel is not mobile. Although cement with a low alkali-and sulphate content were used, DEF and ASR could not be avoided.

After several cycles of climate storage, steel corrosion was also observed at the surface of samples. In final evaluation (2 years storage), steel corrosion and deterioration of the surrounding UHPC matrix were monitored (Figs. 11-13) in mixture B4Q with water storage and intentional pre-damage.

CONCLUSIONS

Investigations of durability of ultra-high performance concrete with the FIB cyclic climate storage test showed that both UHPC mixtures (M2Q-1, B4Q) developed an expansion ‹ 0.4 mm/m (0.4×10^{-3} in./in.) regardless of exposure conditions (WS, HT, or intentional pre-damaging). Results (M2Q-1) in Figure 5 revealed the lowest expansion and highest durability for samples with water storage and without mechanical pre-damage. Heat treatment [90 °C (194 °F)] increased the compressive strength of UHPC but the durability is reduced compared to water stored samples (refer to Fig. 5). Pre-damaged prisms of mixture M2Q-1 possessed sufficient durability.

The increased expansion of mechanical pre-damaged prisms with water storage of mixture B4Q (Fig. 6) is attributed to the steel fiber corrosion (Fig. 11 and 12) and phase changes of the matrix near the steel fibers (Fig. 13). Pre-damaged samples of mixture B4Q with heat treatment show no microstructure changes. The causes for an increased expansion are investigated. The authors did not detect any cracks caused by thermal cooling or autogenous shrinkage which might have an effect on the durability. Examination by electron microscopy of both UHPC mixtures reveal DEF. ASR appears only in the fine-grained UHPC mixture, M2Q-1, due to insufficient dispersed silica fume. The incidence of DEF and ASR does not damage the microstructure and has no negative influence on the durability. Further investigations are necessary to determine potential risk on the durability.

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TABLES AND FIGURES

Table 1—UHPC mixture proportions of Reference 2

	unit	M2Q-1	B4Q
Cement (CEM I 52.5 R HS/NA)	kg/m³	832	650
Basalt	kg/m³	—	597
Quartz sand	kg/m³	975	354
Quartz powder I	kg/m³	207	325
Quartz powder II	kg/m³	—	131
Silica fume	kg/m³	135	177
Steel fibers	kg/m³	192	194
PC I	kg/m³	35.28*	—
PC II	kg/m³	—	30.4*
Water	kg/m³	166	158
W/C (incl. water from PCE)	—	0.227	0.273
slump flow	mm	650	660
Air content	V.-%	2.8	2.6
* 35wt.-% solid content			
Note: To convert from kg/m³ to lb/yd³, divide kg by 0.593.			

Table 2—Chemical composition of the raw materials

	Cement [wt.-%]	Quartz sand [wt.-%]	Quartz powder I [wt.-%]	Quartz powder II [wt.-%]	Silica fume [wt.-%]	Basalt [wt.-%]
CaO	64.1	0	0	0	0.2	10.9
SiO_2	21.8	98.2	98.9	98.9	98.3	41.8
Fe_2O_3	5.1	0.1	0	0	0	11.9
Al_2O_3	3.6	0.9	0.2	0.6	0.4	13.6
K_2O (K_2O_{wsl})	0.36 (0.21)	—	—	—	—	1.30
Na_2O (Na_2O_{wsl})	0.24 (0.06)	—	—	—	—	3.33
SO_3	2.1	—	—	—	—	—
CaO free	0.7	—	—	—	—	—
MgO	—	—	—	—	—	12.8
LOI	1.1	0.1	0.3	1.2	1.3	—

Note: wsl = water soluble

Table 3: Mineralogical composition of cement (according to quantitative XRD-Rietveld analysis)

CEM I 52.5 R HS/NA	
Mineralogical phase	Content (wt.-%]
Alite	62.8
Belite	12.0
Aluminate	2.0
Ferrite	19.8
Gypsum	—
Bassanite	1.8

Table 4: Physical properties of the raw materials

	Unit	Cement	Quartz sand	Quartz powder I	Quartz powder II	Silica fume (SF)	Basalt
Density	[g/cm³]	3.18	2.64	2.64	2.65	2.27	3.07
Spec. surface (Blaine)	[cm²/g]	4900	794	4375	1110	191,000*	—
particle content ‹125µm	[V.-%]	100	1	99.3	63.7	100	—
Medium particle size	[µm]	12.6	351.6	20.1	97.8	0.31	—

Note: particle size distribution obtained by laser diffraction Coulter LS 230.
* spec. surface of SF was determined by nitrogen adsorption (BET).

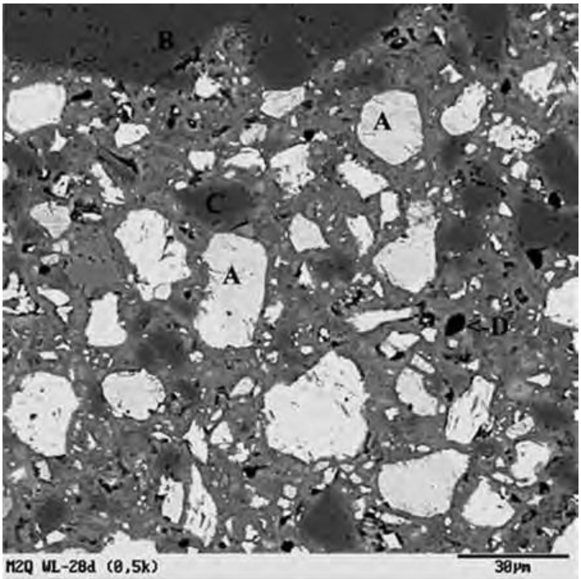

Fig. 1—SEM-BSE image of polished section: Microstructure of fine grained UHPC (W/C = 0.22) at 28 days. (Note: A = incompletely reacted clinker particles, B = quartz sand, C = quartz powder, D = hollows by completely soluted clinker particles).