

LIMITING CONCENTRATIONS FOR ULTRA HIGH STRENGTH CONCRETE IN CONTACT WITH AGGRESSIVE SOLUTIONS

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ABSTRACT

Based on the scientific literature and the results of the priority program “Sustainable Building with Ultra-High Performance Concrete” of the Deutsche Forschungsgemeinschaft (German Science Foundation) the German Committee for Structural Concrete is developing a guideline for Ultra High Strength Concrete (UHSC). One aspect is the durability in contact with aggressive solutions. The development of the guideline is an ongoing process. But some results can be presented, that will probably be accepted by the committee. Although it is widely accepted that UHSC has an increased durability, quantitative data usable within a concept of equivalent performance according to EN 206 are very rare, beginning with the simple question of acceptable damage depths. The contribution presents time dependent damage depths of several UHPC mixtures in contact with different acids and ammonium nitrate solution and other information about chemical attack. In addition the influences of non-static boundary conditions, curing and steel fibres are discussed. Allowed damage depths, measured with a normal strength reference mortar are shown. The results lead to the suggestion that UHSC in contact with aggressive solutions can be used up to the following limiting concentrations without additional protection. Ammonium solutions: 1000 mg/l NH_4^+ , strong dissociated acids: pH 3.5, solutions with aggressive carbon dioxide: until saturation. A prerequisite of this limiting concentrations is that the high strength of the concrete is based on a very low water-cement-ratio and a particle packing optimization. To our opinion the limiting values for sulphate and magnesium solutions can not expanded at the moment, because the data base is too small. Additional research is needed in these cases.

Key-words: UHPC, durability, ammonium attack, acid attack, aggressive carbon dioxide.

INTRODUCTION

In an ongoing process the German Committee for Structural Concrete (DAfStb) is developing a guideline for Ultra High Strength Concrete (UHSC). Of course one aspect is the durability in contact with aggressive solutions. It is widely accepted that UHSC has an increased durability. But increased limiting concentrations for UHSC in contact with various aggressive solutions must be discussed and set, on a scientific basis. Limiting concentrations for exposure classes in the case of chemical attack must be founded on experience from built structures, but such experience does not exist. Alternatively laboratory experiments based on the concept of equivalent performance according to EN 206 [1] can be used. But one very basic difficulty for example is the simple question of acceptable damage depths.

ACCEPTABLE DAMAGE DEPTHS

There are, of course, a great number of papers on chemical attack on concrete. But there is only a very small number of data that can be used to determine a „permissible“ corrosion depth according to the principle of equivalent performance of EN 206 [1]. Concrete under chemical attack (exposure classes XA) is not inert, but shows a time dependent damage. Its rate determines the level of resistance to this environment. Therefore we need information about the „permissible“ corrosion depth as a function of time. The standards do not contain such data, but they can be derived from examinations of equivalent performance. „Permissible“ damage depths can be obtained by corrosion experiments with building

materials in contact with solutions that still are allowed without protective measures according to the standards, and measuring the time dependent corrosion depth. For example Franke et al. [2] found that a damage depth of 0.9 mm is attained after 4000 h (about 5.5 months) for concrete exposed to pH 4.5, the lowest pH value of exposure class XA2 according to EN 206 and the lowest pH concrete according to EN 206 with a w/c-ratio of at least 0.50 may be exposed without protection in Germany. This value includes an additional safety, because the mortar used to determine the damage function had a water-cement-ratio of 0.45, which is already the limit of exposure class XA3. That means also concrete with higher water-cements-ratios are allowed to be used without protective measures in this case. According to [1] a damage depth of 1 mm after 4000 h (5.5 months) is permissible on the basis of EN 206 in the case of acid attack and the resistance of UHSC can be compared with this value.

Hof [3] and Hof et al. [4] reported time dependent corrosion depths of mortars with water cement ratio 0.45 under attack by aggressive carbon dioxide. They amount to 0.6 mm in contact with solution with 60 mg/l aggressive carbon dioxide and 1 mm in contact with solution with 90 mg/l after 1 year. Both concentrations are in the range of exposure class XA2 according to EN 206 [1]. These damage depths are smaller than the damage depths for acid attack, but they were determined with cement paste and not with a mortar that is quite similar to a concrete.

It should be pointed out that a very simple linear extrapolation of permissible damage depths to a normal service life time of 50 years shows that concrete structures under chemical attack may be damaged to a depth of several centimeters during their life. Therefore such damage depths are allowed by EN 206 [1]. But we have to keep in mind, that this is only valid for structures where friction at the surface is not important for the mechanics of the system, but only the residual cross section area, and for non-permeable structures, because permeable structures will have much higher damage depths. And we have to keep this in mind, when construction rules are set for UHSC.

MATERIALS

The results presented in this paper were achieved by experiments with different UHSC of the priority program 1182 of the Deutsche Forschungsgemeinschaft (German Science Foundation), three UHSC from the market without knowledge of the composition (K1, K2, K3) and a normal strength reference mortar (REF). The reference mortar consists of 512 kg/m³ cement CEM I 42.5R-SR, 1536 kg/m³ CEN standard sand and 230.4 kg/m³ water. The water/cement ratio amounts to 0.45. That means this mortar meets the demands of a material according to exposure class XA3 with the exception of the grain size. The water cement ratio of 0.45 is the maximum value that is valid in XA3. The storage of the specimens after the curing took place under water. Table 1 shows the composition of non-commercial UHSC. The additional marking 90 means heat treatment at 90 °C, WL means curing under water.

Table 1 – Composition of the UHSC [5]

	M2Q	B4Q	H75-1
Cement CEM I 52.5R-SR/NA in kg/m ³	832	650	222.5
Quartz sand H33 0.125-0.5 mm in kg/m ³	975	354	1000.9
Basalt 2-8 mm in kg/m ³		597	
Quartz powder W12 in kg/m ³	207	325	221.4
Quartz powder W3 in kg/m ³		131	
Silica fume in kg/m ³	135	177	144.4
Ground granulated blastfurnace slag in kg/m ³			602.9
Steel fibers in kg/m ³	192	192	
Superplasticizer in kg/m ³	10.14 Glenium 51	30.4 Viscocrete20	13.3 Glenium 51
Water in kg/m ³	166	158	179.5

ACID ATTACK

Acid attack leads to a dissolution of the binder (and in the case of acid solubility also of the aggregate) and therefore of the surface. All binder phases may be destroyed, especially the strength building C-S-H-phase of the cementitious material. The damage process is determined by the chemical composition (reactive calcium bearing phases) and the tightness of the pore structure (velocity of the transport processes) of the material.

The following tables show the corrosion depth of different UHSC in contact with acid measured by the proton-consumption method, described in [2] [5] [6] [7].

Table 2 – Corrosion depth in mm with sulphuric acid [5] [6]

	REF	M2Q 90	M2Q WL	B4Q 90	H75-1	K1	K2	K3
pH 3 4000 h	1.57	0.86	0.79	1.11	0.89	1.13	1.05	1.31
pH 3 8000 h	2.41	1.44	1.21	1.65	1.35			
pH 4 4000 h	1.06	0.52	0.46	0.59	0.3	0.49	0.46	0.67
pH 4 8000 h	1.61	0.79		0.91				
pH 5 8000 h	1.16	0.52						

Table 3 – Corrosion depth in mm with lactic acid [5] [6]

	REF	M2Q 90	M2Q WL	B4Q 90
pH 4 4000 h	2.45	1.48	1.33	1.48
pH 4 8000 h	3.09	1.85	1.65	1.88

At pH 3 some UHSC show a higher damage depth than the acceptable depth of 1 mm after 4000 h. At pH 4 all UHSC show a much smaller damage depth than the acceptable one. It can be derived that UHSC in contact with strong dissociated acids with pH 3.5 has an equivalent performance like normal strength concrete in contact with acid with pH 4.5. pH 4.5 is the limiting value for normal strength concretes in contact with acid groundwater without protective measures in Germany by DIN EN 206 [1]. Therefore it seems possible to decrease the limiting value for the pH of strong acids in contact to UHSC to 3.5. In the case of weak acids, the acid capacity must be considered. This can be seen very clear by the data for the lactic acid.

AMMONIUM ATTACK

At high pH-values the ammonium ion NH_4^+ decomposes into gaseous ammonia NH_3 and H^+ , resulting in an attack similar to acid attack, but with a more stable corroded zone. The following table shows the corrosion depths of different UHSC in contact with ammonium solutions measured by the leached calcium. This depth is very similar to the depth of neutralization, determined by the Phenolphthalein-method.

Table 4 – Corrosion depth in mm with ammonium solution with 11250 mg/l NH₄⁺ [5] [6]

	REF	M2Q 90	M2Q WL	B4Q 90	H75-1
4000 h	11.72	4.04	3.54	4.5	2.48
8000 h	15.9	5.7	5.06	6.51	3.56

Based on this data it can be derived that UHSC can be used in the case of ammonium attack without protective measures up to a concentration of at least 1000 mg/l NH₄⁺.

This proposal is also supported by the ATV-guideline 168 [8] (now DWA). Based on this guideline a concrete with a maximum water-cement-ratio of 0.5 will be durable enough in sewer systems with a continuous exposure to an ammonium concentration of 300 mg/l NH₄-N (300 mg/l NH₄-N = 170 mg/l NH₄⁺). An UHSC will have a much higher resistance than such a concrete.

There are many other indications, that the limiting values of EN 206 [1] in the case of ammonium are very low. Bruder [9] noticed, that a concrete test structure with water-cement-ratio 0.5 exposed to ammonium solution with 300 mg/l NH₄⁺ showed no chemical attack. The increase of concentration up to 3000 mg/l NH₄⁺ results only in a slight roughening of the surface of two concrete test structures with water-cement-ratio 0.585 and 0.593 resp. after years of exposure.

Also the results of Rechenberg & Sylla [10] on concrete prisms in ammonium solution after 9 years showed that the actual limiting values of EN 206 are much too safe.

AGGRESSIVE CARBON DIOXIDE

Attack by aggressive carbon dioxide leads to a dissolving of the binder and therefore of the surface, combined with a carbonated zone ahead. Because of the increased tightness and the similarity to acid attack an increased durability of UHSC can be assumed in relation to normal strength concrete. But to our knowledge, there are no data on the durability of UHSC in contact with aggressive carbon dioxide available. Even so it is possible to comment the limiting values for this type of attack. In addition it is important to know, that the maximum concentration of aggressive carbon dioxide is only some hundred mg/l for chemical reasons. That means the saturation concentration is near the lower limiting value of XA3 of 100 mg/l.

Locher & Sprung [11] described a depth of material loss (a parameter that is smaller than the total damage depth) of 2.5 mm after 11 years in aggressive carbon dioxide with appr. 100 mg/l and Locher et al. [12] described a depth of material loss of 6 mm after 20 years (w/c=0.5). Hof [3] described total corrosion depths of hydrated cement stone with w/c=0.45 by aggressive carbon dioxide with 90 mg/l of 1 mm after 1 year. This is about half the „permissible“ damage depth, derived from acid corrosion tests (see above). Also a look into the TGL 11357 [13] of the former German Democratic Republic is very helpful. This very detailed standard classified the attack by aggressive carbon dioxide with more than 90 mg/l as „stark betonaggressiv“ (strong attack to concrete), comparable with an acid attack of pH 5.0 until 4.0, but not as „sehr stark betonaggressiv“ (very strong attack to concrete). On the basis of the arguments named above it is possible to use UHSC in contact with aggressive carbon dioxide without protective measures.

SULPHATE SOLUTIONS

The attack of the sulfate ion on cementitious materials results in the new formation of ettringite and/or gypsum. This chemical reaction needs additional aluminium and calcium. This results in a decrease of the AFm-phase and portlandite. In addition the CaO/SiO₂-relation of the C-S-H-phase can decrease. Most researchers explain the aggressiveness of the sulfate ion in the main by the crystallization pressure of the fine-grained new ettringite in the cement matrix. Some researchers assume a swelling

pressure by water uptake. It is not sure if a new formation of gypsum is also able to destroy real structures, but there are some indications for this. In laboratory experiments a damage process based on the formation of gypsum can be observed. The interaction of a sulfate solution with a concrete structure varies differently dependent on the cation. A solution of potassium sulfate results in expansion and cracks. With additional carbonate, a new formation of thaumasite may occur. In addition, the C-S-H-phase is damaged, so this process is dominated by a loss of strength. The attack of a magnesium sulfate solution is mostly dominated by the magnesium ion, resulting in a strength loss. The damage process is determined by the chemical composition (reactive calcium and aluminium bearing phases), the tightness of the pore structure (velocity of the transport processes) and the strength of the material. [14-23]

Fehling et al. [24] could not detect damages by contact of UHPC with sulfate. Also Schmidt et al. [6] showed that UHPC in contact with sulfate solution will perform better than normal strength concrete. But the differences are in some cases only small. If cracks occur, the increased tightness of the UHPC matrix is no decisive argument for a better performance in comparison to normal strength concrete. Therefore on the basis of the known data it is not possible to introduce an increased limiting concentration for UHPC structures in contact with sulfate solutions at the moment, although there is a potential for such a measure.

MAGNESIUM SOLUTIONS

The magnesium ion is able to replace calcium in its compounds. By this process in cementitious materials a new formation of brucite ($Mg(OH)_2$), hydrotalkite like phases and magnesium silicate hydrates takes place. The C-S-H-phase of the cementitious material will be destroyed. The damage process is determined by the chemical composition (reactive calcium bearing phases) and the tightness of the pore structure (velocity of the transport processes) of the material. During the last years only minor research about the behaviour of cementitious materials in contact with magnesium solutions can be observed, with the exception of magnesium sulfate solutions and magnesium solutions in salt mines [14] [25] [26].

Bentur & Ben-Bassat [25] compared different concrete mixtures with water cement ratio of 0.31 with concrete with a water cement ration of 0.4, in contact with water from the Dead Sea. The brine from the Dead Sea contains in the main magnesium chloride and only small amounts of sulfate. This is of importance for the evaluation, because sulfate itself is an aggressive compound and must be considered otherwise. The reduced water cement ratio resulted in a remarkable increase of the corrosion resistance. UHPC has a smaller water cement ratio than 0.31. Therefore an UHPC in contact with magnesium solution will show an additional increase of corrosion resistance.

But at the moment it is not possible to determine a limiting concentration for the use of UHPC in contact with magnesium solutions. Therefore quantitative time dependent corrosion depths of UHPC at different magnesium concentrations in the solution are necessary. Such data are not known. In Germany the highest magnesium concentration concrete structures can be exposed without additional protective measures is 3000 mg/l (limiting value XA2/XA3). In technical solutions magnesium concentrations of more than 100.000 mg/l are possible. Also natural waters of very high magnesium concentrations are possible. The Dead Sea brine has a magnesium concentration of more than 40.000 mg/l. Data about the damage of UHPC as a function of time in such solutions do not exist. A comparison with a „permissible” time dependent corrosion depth is therefore not possible.

It should be noticed, that the TGL 11357 [13] classified magnesium concentrations above 500 mg/l into the highest aggressiveness, comparable with an acid attack corresponding to a pH-value between 3 and 4. This standard of the former German Democratic Republic was valid also for industrial solutions, not only natural waters like EN 206 [1]. Although UHPC surely is more resistant than normal strength concrete, it is not possible to declare a limiting concentration for UHPC in magnesium solutions without additional protective measures at the moment.

INFLUENCE OF NON-STATIC BOUNDARY CONDITIONS

All the experimental results from [5] and [6] reported above are results of tests with static boundary conditions. In addition corrosion experiments with non-static boundary conditions and sulphuric acid, ammonium solution and sulphate solution were performed. The specimens were immersed into the aggressive solutions for 3 days and dried at 23 °C, 50 % r.h. with an air velocity of 2 m/s for 4 days. This cyclic wetting and drying was repeated up to 36 cycles. Capillary suction is an important transport process in such wetting and drying cycles. Figures 1 and 2 show the results of the experiments with sulphuric acid and ammonium solution.

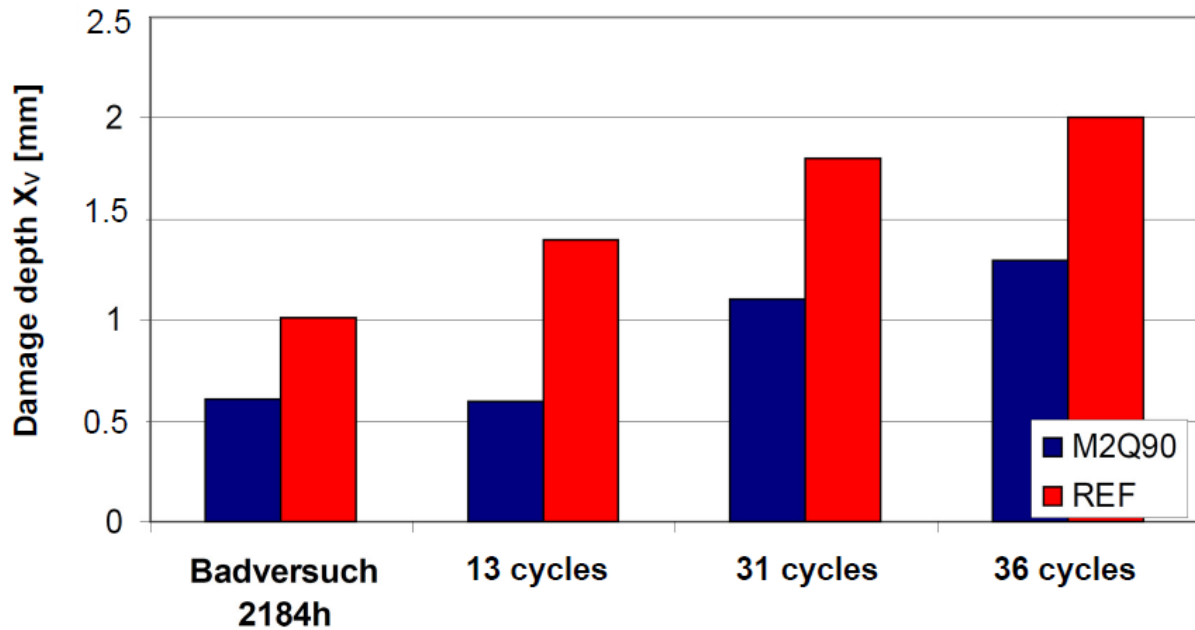


Figure 1 - Damage depth (proton-consumption method) in contact with sulphuric acid, pH3 (ordinate). Comparison of cyclic drying and wetting (cycles) with single and continuous wetting (Badversuch, abscissa). The contact time with the acid at 31 cycles amounts to 2184 h (from [5]).

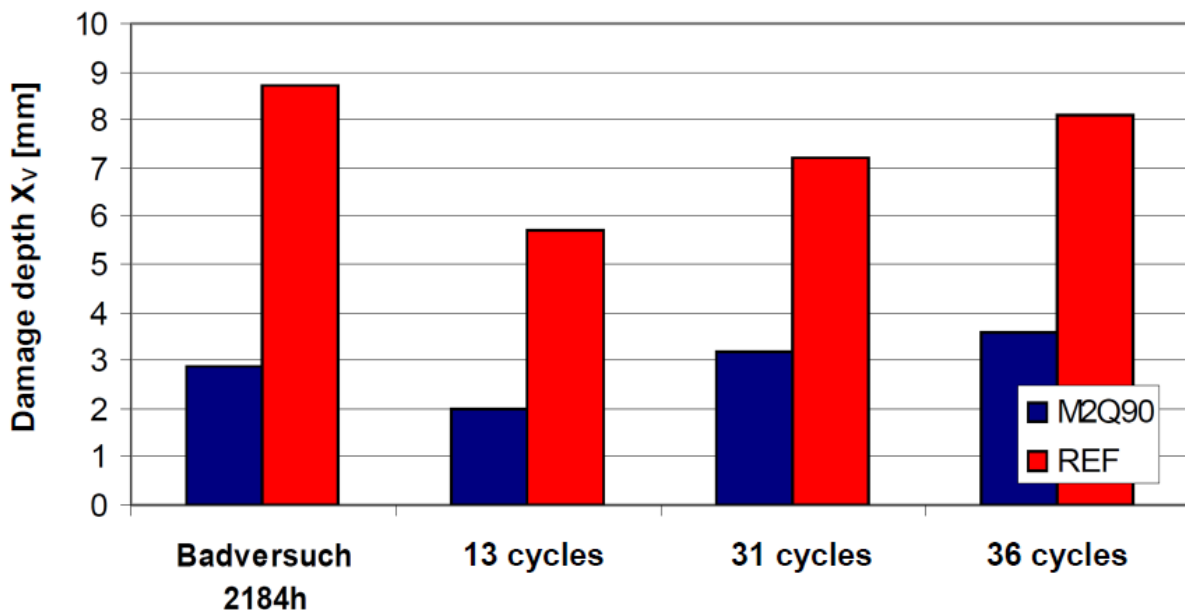


Figure 2 - Depth of neutralization in contact with ammonium nitrate solution (NH_4^+ 11250 mg/l, ordinate). Comparison of cyclic drying and wetting (cycles) with single and continuous wetting (Badversuch, abscissa). The contact time with the solution at 31 cycles amounts to 2184 h (from [5]).

The results can be summarized as follows: „Under cyclic boundary conditions with attack of sulphuric acid a substantially increased corrosion appeared already after few cycles, in comparison to the tests under static boundary conditions. This corresponds to observations in practice. The damage is increased in tidal zones of structures very strongly. The investigation of the attack with sulphuric acid show that the damage depth with cyclic immersion and drying corresponds at least to those of the test with static boundary conditions, although the duration of the contact with the aggressive solution corresponds only to 3/7 of the contact time of the tests with static boundary conditions. By contrast it was found, that the cyclic attack with ammonium nitrate solution, resulting only in dissolution and no new formation of phases, is dependent more or less only on the duration of the direct contact with the aggressive medium." [6]

INFLUENCE OF CURING

The type of curing has a large influence on the durability of UHSC. This can be seen in many of the data in the tables. The specimens with a heat treatment at 90 °C and wrapping in a foil M2Q 90 show higher corrosion rates than the same material without heat treatment but storage under water M2Q WL. It is essential to avoid moisture loss during the heat treatment. Within the priority program 1182 this was later on achieved by weld the specimens into the foil.

INFLUENCE OF STEEL FIBRES

“A significant influence of the steel fibres on the resistance against aggressive media could not be ascertained.” [6]

NEW LIMITING CONCENTRATIONS

In addition to the literature named above a lot of additional papers were evaluated in regard to the topic [27-36], and a great part of the journal Cement and Concrete Research, but no quantitative data to develop limiting concentrations for UHPC in contact with aggressive solutions could be found.

As stated above, the development of the guideline for Ultra High Strength Concrete (UHSC) of the German Committee for Structural Concrete is an ongoing process. The authors propose the following new limiting concentrations for the use of UHSC in contact with aggressive solutions without additional protection in addition to DIN EN 206 [1]: ammonium solutions: 1000 mg/l NH_4^+ , strong dissociated acids: pH 3.5, solutions with aggressive carbon dioxide: until saturation. In the case of weak dissociated acids the acid capacity must be considered. We are optimistic, that this suggestion will be accepted by the committee. It should be emphasized that for UHPC in contact with magnesium or sulfate solutions an increased durability can be expected too. But the quantitative experimental data are missing at this time, to define limiting concentrations for these attacks which allow possible applications of UHPC exceeding EN 206 [1]. Additional research is needed in these cases.

A prerequisite of this limiting concentrations is that the high strength of the concrete is based on a very low water-cement-ratio (lower than 0.25) and a particle packing optimization of the binder. Construction rules for UHSC must keep in mind that in the case of chemical attack there is a time dependent damage of the structure, resulting for example in a loss of material from the surface in the range of centimeters. In the case of chemical attack it is not possible to make structures very thin.

In individual cases UHSC may have a better performance than the limiting values indicate. In this case this individual performance can be proofed by experimental investigations. A test method to determine an individual limiting value in the case of acid attack is described in [2] for example.

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