

# Deformation behavior of concrete due to sulfate attack

## Deformationsverhalten von Beton unter Sulfatangriff

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### 1. Introduction

The phenomenon of mortar and concrete degradation due to sulfate attack is known since more than 100 years. In spite of extensive researches and analyses there are different views about the causes and the mechanisms of action of this important corrosive process. From the scientific point of view, new approaches are therefore required to elucidate completely the manifold dependences as well as to verify the different existing hypotheses. Thus the purpose of this research project was the determination of the chemical, macro- and microstructural input data to develop a model describing the deformation behavior of concrete due the expansive reactions based upon sulfate attack in the pore area and external stress conditions. Within the framework of the collaboration in Research Unit 2 of SPP 1122, sponsored by Deutsche Forschungsgemeinschaft (DFG), numeric analyses should be confronted to extensive experimental microstructure analyses. Thereby the model should be verified and validated as well as possible to couple the sulfate transport in concrete with the mechanical behavior and the damage process.

### 2. Materials and methods

Table 1 gives an overview about the sample spectrum as well as the testing solutions used. Because of time considerations the storage within 0.1 % sulfate solution took place at a temperature of 8°C, because the ettringite formation is accelerated by low temperatures.

Table 1: Sample spectrum overview

Type of samples		Hard. cement paste	Mortar	Concrete
Dimensions of cylindrical samples	[mm]	25 x 25	50 x 50	50 x 50
Dimensions of prisms	[mm]	-	160 x 160 x 40	160 x 160 x 40
Cement content	[kg/m <sup>3</sup> ]	-	450	400
Max. aggregate size	[mm]	-	2	8
Cement type	[-]	CEM I 32.5R CEM I 42.5R CEM I 42.5R-white CEM I 42.5R-HS	CEM I 32.5R CEM I 42.5R CEM I 42.5R-white CEM I 42.5R-HS	CEM I 32.5R CEM I 42.5R CEM I 42.5R-white CEM I 42.5R-HS
w/c-ratio	[-]	0.4 0.5 0.6	0.4 0.5 0.6	0.4 0.5 0.6
Type of solution	[-]	Na <sub>2</sub> SO <sub>4</sub> - 5% Na <sub>2</sub> SO <sub>4</sub> - 0.1% Dist. H <sub>2</sub> O	Na <sub>2</sub> SO <sub>4</sub> - 5% Na <sub>2</sub> SO <sub>4</sub> - 0.1% Dist. H <sub>2</sub> O	Na <sub>2</sub> SO <sub>4</sub> - 5% Na <sub>2</sub> SO <sub>4</sub> - 0.1% Dist. H <sub>2</sub> O

### 3. Experimental procedure

Before storage the samples were checked visually regarding damages. In the course of storage further test-accompanying measurements were conducted. At this all 14 days the heights and masses of the samples subjected to sulfate and water were determined in order to qualify the sulfate solution absorption of the samples and the variation of their water content due to the salt impact. The crack formation and propagation as well as the resulting sample damage on the course of the sulfate weathering were documented by a series of photographs and video recording. The microstructure investigations took place on the basis of an extensive analysis scenario, which combined scanning electron microscopy, particle analysis and energy-dispersive X-ray analysis. The spotlight of the conducted investigations was first the pore system and its ettringite growth as well as its variation on the course of the sulfate attack. Mercury intrusion porosimetry was used as a complementarily analysis for the determination of the influence on the sulfate conditioned expansions to the pore. The investigations took all place at defined time periods, to guarantee the comparability of the results. To support the interpretation of the results, as well as the visualization of the strength reduction in the course of the sulfate attack, compressive and flexural tests were conducted on references and sulfate weathered prisms with dimensions 4 cm x 4 cm x 16 cm.

### 4. Results

**Compressive strength of sulfate stored prisms:** The Compressive strength increases with decreasing w/c-ratio and increasing cement grinding fineness and shows tendentious an increase of the compressive strength during the 56 weathering days independent of the cement type.

**Flexural strength of sulfate stored prisms:** The Flexural strength increases with decreasing w/c ratio and increasing grinding fineness of the cement and shows a constant curve shape during storage independent from the cement type. A decrease of the flexural strength for prisms made of white cement between 56 and 91 days is to be observed.

**Measurements accompanying the storage:** An increase of the w/c ratio leads to higher strains. The concentration of the test liquids has likewise a significant influence on the course of the strain. The specimen stored in a solution with 0.1 % Na<sub>2</sub>SO<sub>4</sub> and the ones stored in water exhibit a similar, almost synchronous strain behavior. For the specimen stored in a solution with 5 % Na<sub>2</sub>SO<sub>4</sub> the initial strain behavior is similar, whereby the duration of this interval depends on the composition of the specimen. A parabolic shaped strain increase follows, which only stops with the destruction of the specimen. The cement type used also determines the characteristic of the strain course. The beginning strain behavior is nearly independent of the cement type and corresponds to the beginning strain behavior described before. An approximately linear and only more slightly strain increase follows in the case of the specimen made of HS cement. In the case of the other cement types it gets in first with one of this dependent latency-time to the parabolic shaped increase of strain ending with the destruction of the specimen as described before. Clear differences in the strain course between hardened cement paste, mortar and concrete specimen made of the same cement type and the same w/c ratio appear. A significant accelerating strain increase was already to be observed after 90 days on hardened cement paste specimen made of white cement with a w/c ratio of 0.50 after a weathering with a solution with 5 % Na<sub>2</sub>SO<sub>4</sub>. In comparison with this, following the initial strain, the mortar and concrete specimen of the same w/c ratio showed up a largely linear, but only weakly pronounced increase of strain, which is larger for the mortar specimen than in the case of concrete, over the entire storage period of 400 days.

**Porosity:** The overall porosity of the hardened cement paste samples decreases faster than those of mortar and concrete due to closing of the pores with the crystal growth due the effect of the Na<sub>2</sub>SO<sub>4</sub>-solution. An increasing porosity within the investigation period indicates the beginning of a sample destruction caused by the increasing crystallization pressure. This phenomenon occurs much earlier in the case of the hardened cement paste specimen than in the case of the mortar and concrete specimen. Analogously, the development of the pore size distribution proceeds to it. In the case of the hardened cement paste specimen it shifts the fastest towards smaller pore radii in the course of storage.

**Ettringite growth:** Very varying ettringite shapes appear in dependence of the different parameter combinations. Sphere and pin shaped ettringite whereas non-directional and directional growth can be observed in the latter. The velocity and the intensity of the new formation of ettringite are dependent on the cement type, whereas

in each case the specimen made of white cement showed the fastest ettringite growth due to their comparatively higher C<sub>3</sub>A content, which correlates to the statements regarding crack, strain and porosity development. The higher the water cement ratio, the more rapidly and more intensively the process of ettringite new formation takes place. It can be observed the higher the sulfate concentration of the weathering solution, the higher the ettringite growth velocity. The size of those pores, in which ettringite growth is observed, is reduced with increasing storage duration. The ettringite growth takes mainly place in pores with a size starting from 10 µm. No ettringite growth was observed in pores within the nanometer range. The new formation of ettringite and the accrue of the pores in the case of hardened cement paste specimen take place substantially faster than for mortar and concrete. The slowest ettringite growth was to be observed at concrete specimen.

## 5. Outlook

The following points are of interest regarding the future research requirement:

- Apart from the past investigations regarding the growth of ettringite in the pore volume of the microstructure of concrete is to be aimed to also identify the various phases of the bonding agents as well as to characterize the spatial arrangement of the pore system and the solid structural constituents, in order to be able to make statements about variations of the microstructure and about the variation of the ratio matrix, pore system and clinker residues in the course of sulfate weathering.
- Since the transport and crystallization processes primarily take place in the capillary pore volume, the recording of its physical structure is a substantial condition both for the micromechanical observations to local deformations on the micro scale as well as to macroscopic structural variations, which in particular concern the cracking and the crack propagation processes. By the gradual uptake of defined sample sections in combination with the following degradation of the sample surface in each case so called image field stacks can be generated, which step instead of individual pictures and so make the transition possible from the two-dimensional image of the structure to the three-dimensional record and representation of the microstructure.
- Further clarifying requirement exist due to the lack of sufficient knowledge about the cause and the effect of the occurrence of varying ettringite formations.