PRACTICAL INVESTIGATION OF THE SULFATE RESISTANCE OF CONCRETE FROM CONSTRUCTION UNITS

PRAXISNAHE UNTERSUCHUNG DES SULFATWIDERSTANDES VON BETON AUS BAUTEILEN

ETUDE PRATIQUE DE RESISTANCE DU BETON DES ELEMENTS SOUS ACTION DE LE SULFATE

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SUMMARY

To determine the sulfate resistance of a sports hall foundation concrete samples (drill-out cores) were taken out of the foundation structures for being compared with lab-manufactured concretes and mortars under varying conditions. Following here the so called flat prism procedure, samples (drill-out cores) from the new building and different composed types of concrete drill-out cores as well as flat mortar prisms were immersed in a sodium sulfate solution (29800 mg sulfate/l). The expansion of the specimen were measured up to 150 days storage as well as 540 days of a long-term storage.With regard to the temperature conditions of the foundation in situ the tests have been carried out not only at 20 °C but also under more severe conditions at 6 °C.

ZUSAMMENFASSUNG

Zur Bestimmung des Sulfatwiderstandes eines Fundamentbetons einer Sporthalle wurden aus den Gründungskörpern des Neubaus Bauteilproben (Bohrkerne) entnommen und mit labormäßig hergestellten Betonen und Mörteln unter variierten Lagerungsbedingungen verglichen. Hierbei wurden in Anlehnung an das sogenannte Flachprismenverfahren Bauteilproben (Bohrkerne) des Neubaus der Sporthalle und unterschiedlich zusammengesetzte Betonbohrkerne und Mörtelflachprismen in Natriumsulfatlösung (29800 mg Sulfat/l) gelagert und die Dehnungen bis zu einer Lagerungsdauer von 150 Tagen sowie nach 540-tägiger Langzeitlagerung bestimmt. Mit dem Ziel die Temperaturverhältnisse im Gründungsbereich des Neubaus der Sporthalle praxisnah abzubilden, wurden die Versuche nicht nur bei 20 °C, sondern auch unter verschärften Bedingungen bei 6 °C durchgeführt.

RESUME

Pour déterminer la résistivité de sulfate d´un béton de fondations d´une salle omnisports des éléments échantillons (centres d´enfonce) étaient prélevés des corps de fondation de la nouvelle construction, et les étaient comparés avec les centres d´enfonce et prismes du mortier fabriqués en laboratoire dans des conditions de conservation variers. Les éléments échantillons (centres d´enfonce) de la nouvelle construction de la salle d´omnisports et des centres d´enfonce du béton composés divers et prismés du mortier étaient montés dans une solution du sodium sulfate à 4,4 pour cent suivant l´exemple du procédé des prismes, et les extensions étaient déterminées jusqu´à une durée de conservation des 150 jours et après une conservation de longue durée des 540 jours. Les épreuves étaient exécutées aux 20 °C et aussi aux 6 °C dans des conditions plus sévères, avec le but de reproduire les conditions de température axé sur la pratique en domaine de zone de fondation d´une nouvelle construction d´une salle omnisports.

1. INTRODUCTION

At the end of the construction of a new building of a sports hall the responsible city administration contacted the Otto-Graf-Institut of the MPA University of Stuttgart. Because of contradictory test results it was unclear if the required compressive strength of the foundation of the sports hall was reached or not. Furthermore a cracking of the concrete floor and an apparently defective concrete surface were criticised. In addition to this the resistance of the foundation concrete was to be investigated in relation to strong sulfate attack because the present groundwater contained significant amounts of sulfate and the utilised water/cement ratio (w/c-ratio) was not in accordance with relevant standards.

2. REGULATIONS CONCERNING SULFATE ATTACK ON CONCRETE

Concrete units exposed to sulfate attack may be damaged by expansion by the cause of the formation of secondary ettringite and secondary gypsum [1]. At low temperatures predominating in foundation structures it is known that under unfavorable conditions an additional damage may occur by the formation of thaumasite. To protect a secure production method of concrete building, concrete which is exposed to a sulfate attack must have a high resistance against chemical attack according to DIN 1045 [2] and DIN EN 206-1 [3]/ DIN 1045-2 [4] respectively. In compliance with the regulations no damages occured so far [1].

The resistance of concrete against sulfate attack consists of a chemical and a physical resistance. For chemical resistance the used cement and/or the used cement-fly ash combination is relevant. For physical resistance the microstructure density is significant which is usually controlled by a suitable w/c-ratio.

2.1 Current State of Standardization

According to DIN 1045 [2], chapter 6.5.7.5, the resistance of the concrete structure against chemical attack depends basically on its density. The w/c-ratio of the concrete may therefore not exceed by „weak“ attack 0.60 and 0.50 by „strong“ attack. Further a cement with high sulfate resistance, according to DIN 1164-10 [5], must be used if the sulfate content exceeds 600 mg per litre water, without seawater present. In case of foundations the threshold value is 3000 mg sulfate per kg soil.

The concrete standard DIN EN 206-1 [3]/DIN 1045-2 [4] which must be used starting from 01.01.2005 classifies concretes depending on the ambient conditions in exposure classes. Thereby the exposure classes for concretes exposed to chemical attack by natural soil, groundwater, sea water and waste water are assigned as followed:

- XA1: chemically weak corrosive
- XA2: chemically moderate corrosive
- XA3: chemically strong corrosive

The particular requirements of the exposure classes XA1, XA2 and XA3 are represented in table 1.

Tab. 1: Measures for the production of concrete with high sulfate resistance against water containing sulfate.

	Aggressive surrounding area		
	chemically weak	chemically moderate	chemically strong
Exposure classes	XA1	XA2	XA3
Criterion for SO_4^{2-} in mg/l in groundwater	≥ 200 and ≤ 600	> 600 and ≤ 3000	> 3000 and ≤ 6000
Max. w/c-ratio	0.60	0.50	0.45
Minimum compressive strength class	C 25/30	C 35/45[1)] (C 30/37[1)])[2)]	C 35/45[1)]
Minimum cement content in kg/m³	280	320	320
Minimum cement content by charging for additives in kg/m³	270	270	270

1) by use of air-entrained concrete, for example as result of coexistent requirements from the exposure class XF, one compressive strength class lower

2) according to the draft to the A1 amendment of the DIN 1045-2 [6] a reduction of the compressive strength is intended

2.2 Cements with High Sulfate Resistance

Cements with high sulfate resistance (HS-cements) must meet the requirements of DIN EN 197-1 [7] and DIN 1164-10 [5]. Blast furnace cement CEM III/B is considered as a HS-cement with high sulfate resistance due to its high granulated blast furnace slag content (≥ 66 % by mass).

2.3 Influence of Additives on the Sulfate Resistance

The sulfate resistance of blast furnace cements increases with granulated blast furnace slag contents above approximately 60 % by mass to high values [8]. Reason for this is the high diffusion resistance of the hardened blast furnace cement structure at higher slag contents which cannot be penetrated by sulfate ions. Starting from a granulated blast furnace slag content of at least 65 % by mass an increase of the granulated blast furnace slag content affects the increase of the sulfate resistance substantially more than a reduction of the w/c-ratio [8].

The increase of the sulfate resistance by the puzzolanic effect of hard coal fly ash is basically attributed to higher concrete tightness and the guarantee of improved diffusion resistance against sulfate ions by additional formation of CaO-poorer C-S-H-phases [8].

3 TESTING METHODS FOR THE DETERMINATION OF THE SULFATE RESISTANCE

3.1 Testing Methods for Cement

For the determination of the sulfate resistance of cement there are different national and international testing methods common. In the majority the acceleration of the sulfate entry into the samples is accomplished by storing in very high sulfate concentrations. These concentrations are partially ten times higher than the accepted sulfate concentrations in practice. The assessment of the majority of testing methods is done by measuring the expansion of flat prisms during sulfate storage and reference storage which are compared with a predefined criterion. In addition to common test methods by expansion-measurements the test method of Koch-Steinegger [8] as well as the MNS procedure [9] are practiced in Germany. It is not the intention of this article to deal with these testing methods in greater detail. Table 2 shows a comparison of the accelerated test methods by expansion measurements according to Wittekindt [10], SVA [8] and CEN [8].

Tab. 2: Comparison of accelerated test methods by means of expansion measurement

	Wittekindt	SVA	CEN
Test pieces	10 x 40 x 160 mm^3	10 x 40 x 160 mm^3	20 x 20 x 160 mm^3
w/c-ratio	0.60	0.50	0.50
Sulfate solution (Na_2SO_4)	4.4 % (29800 mg sulfate/l)	4.4 % (29800 mg sulfate/l)	2.4 % (16000 mg sulfate/l)
Storage period	56 days	91 days	not defined
Criterion	≤ 0.50 mm/m	≤ 0.50 mm/m	not defined

2.2 Testing Methods for Mortar and Concrete

At present the evaluation of the sulfate resistance of concrete is still subject of research. The todays used testing methods for concrete orientate at the testing methods for cement and are performed in this or in modified ways [9].

In Germany the determination of the sulfate resistance of mortar and concrete is carried out with several methods. In order to determinate the sulfate resistance of concrete the testing method of the committee of experts (SVA) of the German Institute for Civil Engineering appeared to be suitable in this case.

In this testing method flat prisms according to DIN EN 196-1 [11] are manufactured with a w/c-ratio = 0.50 and are cast in prisms 10 x 40 x 160 mm^3. The specimen are demoulded after 2 days storage in moist air and are additionally stored 12 days in saturated $Ca(OH)_2$-solution. The following storage takes place in a 20 °C sodium sulfate solution (29800 mg sulfate/l). The solutions are monthly renewed. The sulfate resistance is rated by the expansion difference ($\Delta\varepsilon \leq$ 0.50 mm/m) after 91 days (=HS-criterion).

4 TEST PROGRAM

4.1 Intention

The investigations are focused on two aspects. On the one hand the expansion behavior should be compared with mortar prisms that were produced with a binder combination of blast furnace cement and fly ash and were stored in a sodium sulfate solution (29800 mg sulfate/l). By the way the binder components of the foundation concrete and the mortar prisms derived from the same origin. Apart from the usual storage temperature of 20 °C the samples were also stored at 6 °C which is regarded as an intensification of the test conditions. On the other hand there is the question what effect an increase of the acceptable w/c-ratio has on the structure density and thus on the resistance of the foundation structure in regard to sulfate attack. For this reason mortar prisms with different cement types and varying w/c-ratio were examined using the flat prism test. In addition to this samples from the foundation concrete were compared to standard manufactured concrete specimen produced in accordance to DIN 1045 [2] and DIN EN 206-1 [3]/DIN 1045-2 [4] respectively.

4.2 Mortar Tests according to the Flat Prism Procedure

Following the flat prism procedure three mortar mixtures were produced. The w/c-ratio of the mixtures 1 and 2 meets the guidelines of the flat prism procedure. For the mixture 3 the w/c-ratio of 0.68 was selected according to the composition of the foundation concrete in situ. The mortar compositions are presented in table 3:

Tab. 3: Mortar composition of the flat prisms, mixture 1 - 3

Description	Mixture 1	Mixture 2	Mixture 3
Cement type Cement content in g	Portland cement CEM I 32.5 R 450.0	Blast furnace cement CEM III/B 32.5 – NW/HS 420.5	Blast furnace cement CEM III/B 32.5 – NW/HS 420.5
Fly ash content in g	–	73.8	73.8
Water content in g	225	225	306
w/(c + k·f) k = 0.4	0.50	0.50	0.68

The mixed mortar was cast in the moulds for flat prisms with the dimensions 10 x 40 x 160 mm^3 and was compacted on the vibration table according to DIN EN 196-1 [11]. A two-day storage of the prisms followed at 20 °C in moist air of ≥ 90 % relative humidity. Subsequently the specimen were demoulded and were stored 12 days in saturated $Ca(OH)_2$-solution. After this the initial length was measured. The following storage conditions subdivide into:

- 20 °C in saturated $Ca(OH)_2$-solution (reference solution)
- 20 °C in sodium sulfate solution (29800 mg sulfate/l) (test solution)
- 6 °C in saturated $Ca(OH)_2$-solution (reference solution)
- 6 °C in sodium sulfate solution (29800 mg sulfate/l) (test solution)

The length change of the specimen was measured at 14, 28, 56 and 91 days. A replacement of the Na_2SO_4-solution was carried out in fortnights.

4.3 Concrete Tests following the Flat Prism Procedure

At first concrete samples (drill-out cores ∅ = 50 mm) from the foundation structure of the sports hall building were taken. The composition of the utilised concrete is shown in table 4:

Tab.4: Composition of the sports hall foundation concrete

Description	S IV (construction units)
Cement type Cement content in kg/m^3	Blast furnace cement CEM III/B 32.5 – NW/HS 285
Fly ash content in kg/m^3	50
Water content in dm^3/m^3 w/(z+0.4·f)	207.4 0.68
Aggregates Grading curve Content (dry) in kg/m^3	sand, shell-limestone A/B 22 1771

Furthermore three additional concretes were produced for comparison (S I, S II and S III). Their composition is shown in table 5:

Tab. 5: Composition of the comparison concrete S I, S II and S III

Description	S I	S II	S III
Cement type Cement content in kg/m^3	Portland cement CEM I 32.5 R 305	Blast furnace cement CEM III/B 32.5 – NW/HS 285	Blast furnace cement CEM III/B 32.5 – NW/HS 285
Fly ash Content in kg/m^3	–	50	50
Limestone meal Content in kg/m^3	30	–	–
Water content in dm^3/m^3 $w/(z+0.4 \cdot f)$	207.4 0.68	183.0 0.60	152.5 0.50
Aggregates Grading curve Content (dry) in kg/m^3	S/M[1)] A/B 22 1746	S/M[1)] A/B 22 1792	S/M[1)] A/B 22 1871
Concrete admixtures Content in ml/kg[2)]	– –	– –	Superplasticizer 7

[1)] sand, shell-limestone
[2)] related to the cement content

For each concrete mix S I, S II and S III three cubes with 200 mm of edge length were cast. The cubes were stored after the production at 20 °C in moist air of ≥ 95% relative humidity. At the age of two days four drill-out cores ∅ = 50 mm were taken from each cube.

Before immersion the lab manufactured drill-out cores were sawn to a length of 150 mm and were flat polished at the top surfaces similar to the construction samples (S IV). Furthermore stain plugs were fixed at the surface to measure length variation. Until the initial measurement the drill-out cores were stored at 20 °C and 65 % relative humidity for 12 days.

Following the flat prism procedure (see section 3.2) in each case three drill-out cores of the concretes S I, S II, S III and S IV (construction samples) were immersed at 20 °C in a sodium sulfate solution (test solution) as well as in water (reference solution). According to [8] the flat prism procedure at 20 °C storage temperature doesn't always sufficiently represent the environmental conditions. For this reason three drill-out cores of the concretes S I, S II, S III and S IV were stored at 6 °C sodium sulfate solution and in water additionally. The immersion at 6 °C is regarded as a more severe test condition in comparison to 20 °C storage temperature.

The length change of the specimen was measured at 0 (initial value), 2, 5, 7, 14, 28, 56, 91, 120 and 150 days of immersion. The sodium sulfate solution was also changed in fortnights.

For the determination of the long-term behaviour the drill-out cores of the comparison concrete S I and the drill-out cores from the building (S IV) were finally measured after 540 days beyond the regular storing range.

5 RESULTS

5.1 Results on Mortar Prisms

To quantity the sulfate resistance of the specimen the expansion of ≤ 0.50 mm/m after 91 days immersion was chosen as a threshold value. Fig. 1 and 2 show the expansion behaviour of flat mortar prisms stored in a 20 °C and 6 °C sodium sulfate solution (29800 mg sulfate/l).

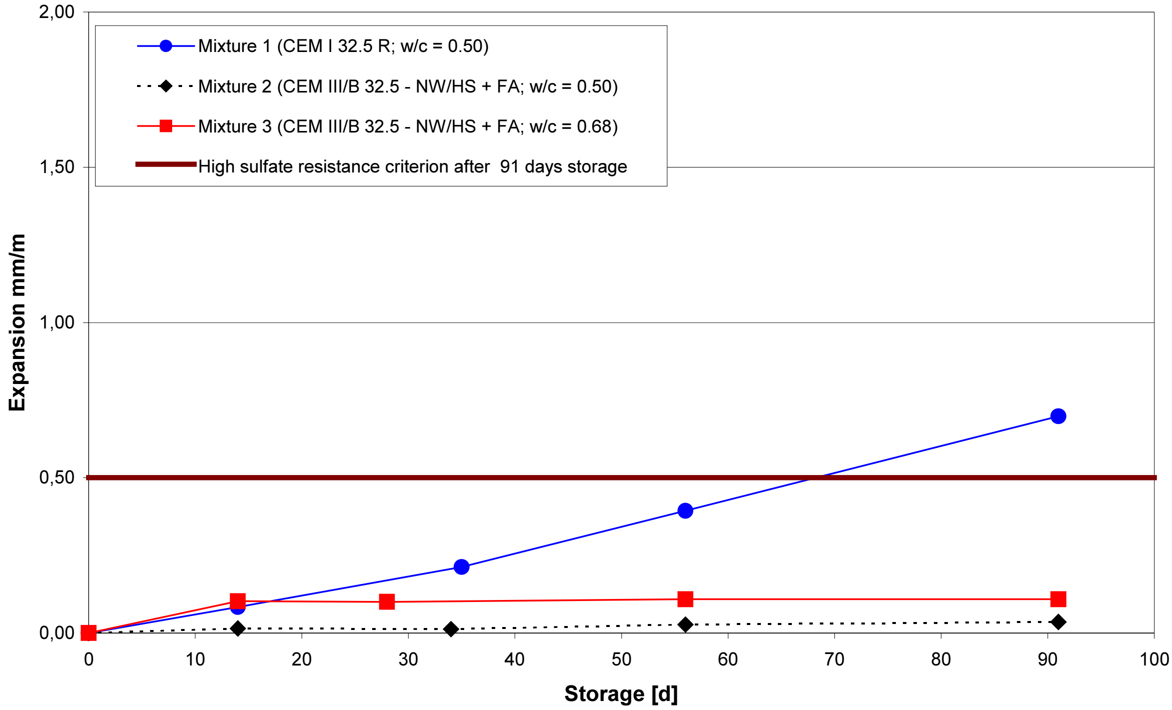

Fig. 1: Expansion behaviour of flat mortar prisms immersed in sodium sulfate solution (29800 mg sulfate/l). Storage temperature 20 °C

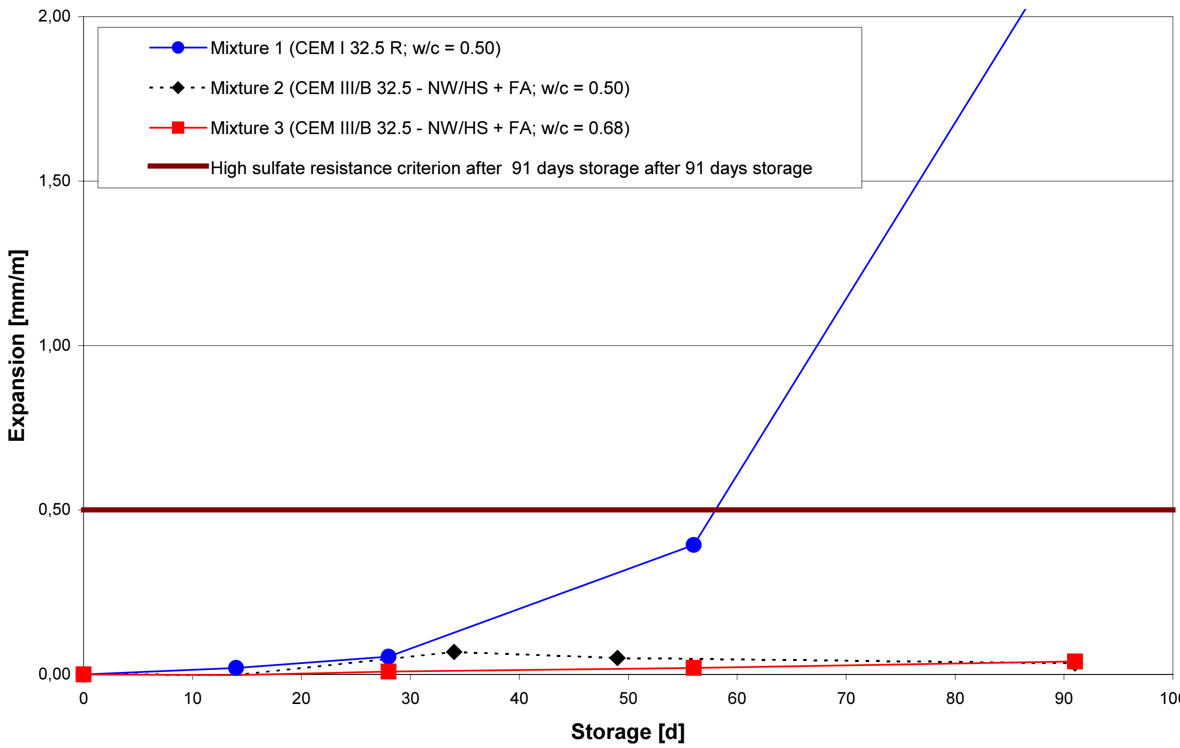

Fig. 2 : Expansion behaviour of flat mortar prisms immersed in sodium sulfate solution (29800 mg sulfate/l). Storage temperature 6 °C

The mortar prisms of the mixtures 2 and 3 passed the high sulfate resistance criterion both at 20 °C and 6 °C after 91 days storage in sodium sulfate solution. The prisms of mixture 1 did not fulfill the high sulfate resistance criterion both at 20 °C and 6 °C sodium sulfate solution after 91 days storage as expected.

5.2 Results on Concrete Specimen

The threshold expansion of ≤ 0.50 mm/m after 91 days from chapter 5.1 was also taken to assess the resistance of concrete specimen against sulfate attack. Fig. 3 and 4 show the expansion behaviour of concrete drill-out cores immersed at 20 °C and 6 °C in a sodium sulfate solution.

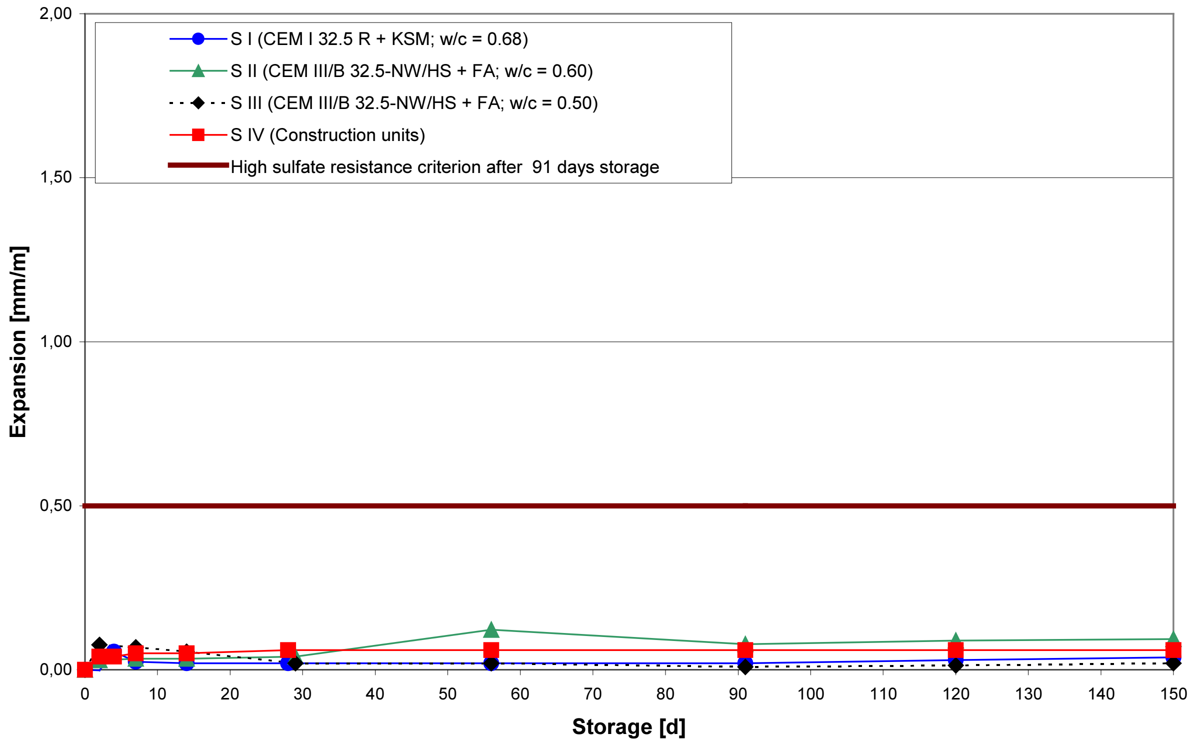

Fig. 3: Expansion behaviour of concrete drill-out cores immersed in sodium sulfate solution (29800 mg sulfate/l). Storage temperature 20 °C

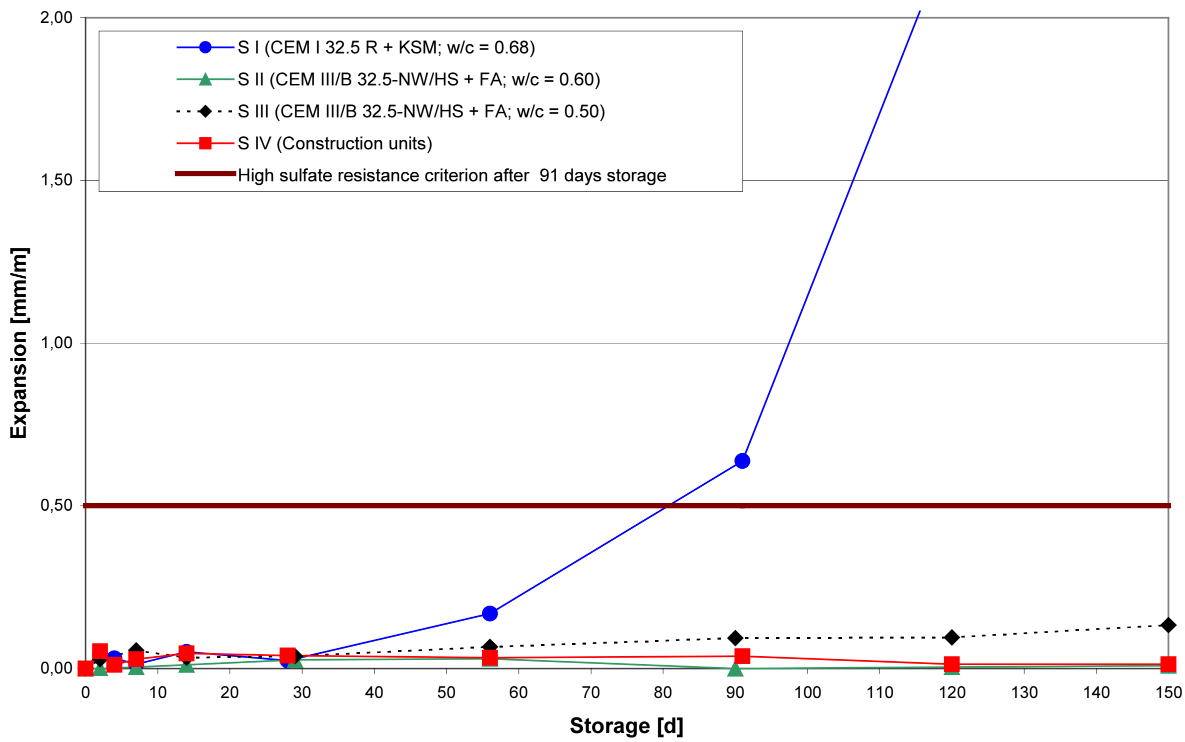

Fig. 4: Expansion behaviour of concrete drill-out cores at storage in sodium sulfate solution (29800 mg sulfate/l). Storage temperature 6 °C

The drill-out cores from the comparative concretes (S I, S II, S III) and the drill-out cores taken from the building (S IV) fulfilled the high sulfate resistance criterion of the flat prism procedure at the storage temperature of 20 °C. The measured expansion after 150 days immersion in a sodium sulfate solution (29800 mg sulfate/l) were significantly lower as the threshold value of 0.50 mm/m.

However the drill-out cores from the comparison concrete S I showed a very high expansion at 6 °C and did not meet the criterion for a high sulfate resistance. The other test specimen S II and S III as well as the samples taken from the sports hall concrete presented no significant expansion increase and were not damaged even after a period of 150 days storage.

The expansion of the specimen from comparison concrete S I and the drill-out cores derived from the building (S IV) have also been stored in a long term test for 540 days. Table 6 shows the results after 540 days of immersion.

Tab. 6: Expansion behaviour of the drill-out cores S I and S IV after a storage duration of 540 days. Immersion in sodium sulfate solution at 6 °C and 20 °C.

Immersion time	S I		S IV	
[d]	20 °C	6 °C	20 °C	6 °C
150	0.04 mm /m	destroyed	0.06 mm/m	0.01 mm/m
540	3.46 mm/m	destroyed	0.06 mm/m	0.07 mm/m

Even after 540 days in a 20 °C and 6 °C sodium sulfate solution the drill-out cores from the building (S IV) have still shown a low expansion far beneath the threshold value of 0.50 mm/m. Furthermore no damages were visible in form of cracks or spallings. On the other hand the expansion of comparison concrete S I lay clearly above the threshold value of 0.50 mm/m after 540 days storage in a 20 °C sodium sulfate solution while an immersion at 6 °C has led to total disruption.

Fig. 5 (left) shows the specimen at the beginning and fig. 5 (right) after a storage duration of 150 days. The complete destruction of the drill-out cores S I is shown in fig. 5 (right).

Fig. 5: Concrete drill-out cores at the beginning of the test (left picture) and destroyed drill-out cores S I (right figure) after 150 days immersion.

The destroyed specimen from concrete S I, see fig. 5 (right), show a classical damage by sulfate attack. The damages mainly occurred due to the formation of secondary ettringite and secondary gypsum. In addition to this sulfate attack can also lead to a destructive formation of thaumasite, which weakens the microstructure at low temperatures (< 15 °C) [12]. All three causes for damage by sulfat attack were detected by scanning electron microscope (fig. 6) and x-ray-diffraction (fig. 7).

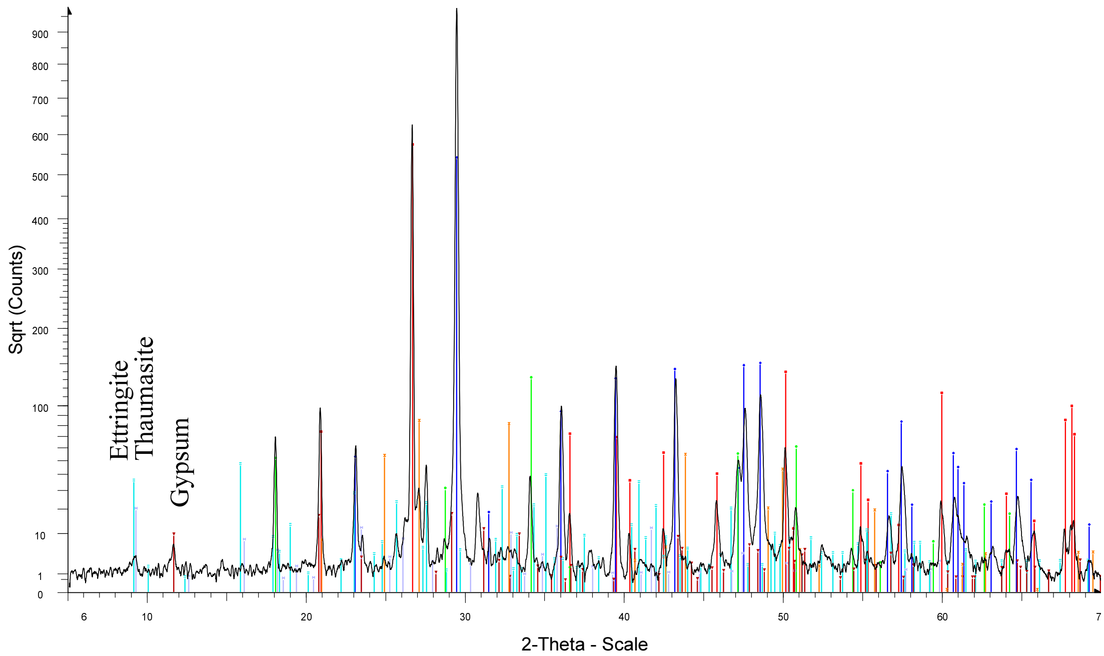

Fig. 6: X-Ray-Diffraction exposure of a destroyed concrete section (S I) after 150 days immersion in sodium sulfate solution (29800 mg sulfate/l). Storage temperature 6 °C

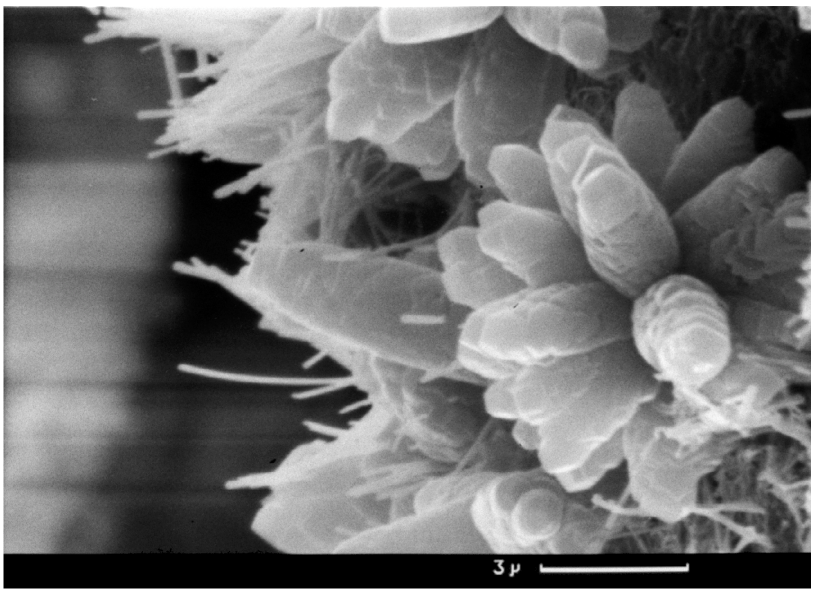

Fig. 7: Scanning electron microscope – Secondary ettringite and secondary gypsum of a concrete section (S I) in the crack area after 150 days immersion in sodium sulfate solution (29800 mg sulfate/l). Storage temperature 6 °C

6 SUMMARY AND EVALUATION OF THE RESULTS

To determine the sulfate resistance of a sports hall foundation concrete samples (drill-out cores) were taken out of the foundation structures for being compared with lab-manufactured concretes and mortars under varying conditions. Following here the so called flat prism procedure, samples (drill-out cores) from the new building and different composed types of concrete drill-out cores as well as flat mortar prisms were immersed in a sodium sulfate solution (29800 mg sulfate/l). The expansion of the specimen were measured up to 150 days storage as well as 540 days of a long-term storage. With regard to the temperature conditions of the foundation in situ the tests have been carried out not only at 20 °C but also under more severe conditions at 6 °C.

In particular, the tests have shown that:

- Concrete cores made with a blast furnace cement/fly ash mix with a w/c-ratio of 0.50, 0.60 as well as 0.68 (foundation concrete) met the high sulfate resistance criterion of the rapid test in a 20 °C and 6 °C sodium sulfate solution (29800 mg sulfate/l) after 91 days of immersion. The test criterion was even achieved after 150 days storage.
- Concrete cores made with mixtures of blast furnace cement and fly ash with a w/c-ratio of 0.68 (foundation concrete) fulfilled the high sulfate resistance test criterion in a 20 °C and 6 °C sodium sulfate solution after a 540 days long-term storage.
- Concrete cores made with mixtures of ordinary Portland cement and limestone-meal did not meet the requirement of the test criterion in a 20 °C sodium sulfate solution after the 540 days long-term storage.
- Flat mortar prisms made with mixtures of blast furnace cement and fly ash with a w/c-ratio of 0.50 and 0.68 fulfilled the high sulfate resistance test criterion in 20 °C as well as in 6 °C sodium sulfate solution after a 91 days storage.
- Flat mortar prisms made with ordinary Portland cement showed a significantly lower sulfate resistance at 6 °C as well as at 20 °C and did not meet the high sulfate resistance criterion of the flat mortar procedure.

Due to the similar results of the concrete and mortar investigations at a storage temperature of 6 °C it can be assumed that the high sulfate resistance criterion from the flat prism procedure is comparable with the concrete investigations. It can also be concluded that the concrete of the sports hall made with a unduly w/c-ratio of 0.68 showed a similar behaviour under sulfate attack as concrete in accordance to relevant standards.

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