



Does gypsum formation during sulfate attack on concrete lead to expansion?

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Abstract

Sulfate attack on Portland cement concrete is often said to arise from each of two major sulfate reactions: (1) The sulfate ions react with C_3A and its hydration products to form ettringite with an increase in volume that results in expansion and subsequent cracking of the concrete; (2) The sulfate ions react with calcium hydroxide (CH) to form gypsum. Even though gypsum formation is generally accepted to be harmful, the specific mechanism is not well established. Especially, the idea that gypsum formation leads to any expansion is controversial. This paper covers an investigation carried out to study the gypsum formation during sulfate attack and its consequences. Two parts are included: Part 1 consists of the results of a literature review describing different theories supporting and contradicting the idea that gypsum formation is expansive. Part 2 describes the laboratory investigation carried out by the authors. The results suggested that gypsum formation during sulfate attack may cause expansion. © 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Sulfate attack is the destructive process acting on concrete due to the formation of expansive reaction products within concrete exposed to external sulfate sources. The widespread occurrence and destruction caused by sulfate attack attracted researchers over the years into the study of the deterioration mechanism and methods to combat it. However, different theories about the mechanism still exist and the mechanism is not well understood. One of the established findings so far is the understanding of the damaging effect of C_3A or other chemically active sources of aluminate in the concrete, such as aluminate-bearing glass in fly ash, that are responsible for ettringite formation. Ettringite is an expansive product formed within concrete. It is believed by some researchers to be the only source of expansion during sulfate attack. Expansion can lead to cracking, spalling, and other damaging effects. The damage mechanism of ettringite has been summarized into two schools of thought [1]. Based on the widely accepted finding that a correlation exists between the C_3A content in Portland cement and the susceptibility of concrete to sulfate attack, ASTM Type II and V cements, having limited C_3A content, were introduced to the market. These cements are normally

used in the concrete that is exposed to moderate and severe sulfate concentrations. Nonetheless, in some field cases and laboratory reports it was reported that the use of Type V cement, with the amount of C_3A less than 5%, may not prevent damage due to sulfate attack [2,3]. Moreover, in some cases, use of zero- C_3A cement may not provide resistance to sulfate attack [3]. These data indicate that the sulfate-attack mechanism is indeed complicated and expansion and cracking of Portland cement concrete under sulfate attack should not exclusively be attributed to ettringite formation. Other factors, especially gypsum formation, should also be taken into consideration as a possible source of expansion. However, research specifically designed to study the damaging effects and mechanism of gypsum formation during sulfate attack is quite limited. There are more suggestions that are not supported by experimental data than there are facts obtained from experimental work. The studies reported have normally been limited to Portland cement concrete that contains aluminate phases. The expansion is attributed to ettringite formation. It is often accepted that the gypsum formation only has a softening effect and causes mass and strength loss [4]. There is no clear documentation supporting that gypsum formation leads to expansion.

The study of the role of gypsum formation in sulfate attack not only is important to the understanding of the sulfate-attack mechanism, but also provides evidence for the adoption of a limitation on C_3S content of Portland cement.

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Day and Joshi [5] predicted that sulfate attack on concrete would become more serious with increasing C_3S content in modern cements. An increase of CH generated during the hydration of Portland cement may consequently permit the production of a large amount of gypsum in concrete exposed to external sulfate sources, and the subsequent damage could be serious.

In this paper, the authors first reviewed the literature dealing with the deterioration and mechanism of sulfate attack caused by gypsum formation. In the next section, the experimental investigation is introduced and discussed. Alite paste specimens were exposed to Na_2SO_4 solution and $(NH_4)_2SO_4$ solution. C_3S mortar specimens were exposed to Na_2SO_4 solution. The physical properties and chemical reactions were studied to determine if gypsum formation is associated with expansion.

2. Literature review

Until now there has been no clear documentation that gypsum formation associated with sulfate attack leads to expansion. The theories either supporting or contradicting this idea are not well established. The following is a literature review with emphasis on the publications of recent years. The literature was categorized into three sections: those contradicting the idea that the gypsum formation is expansive, those supporting the idea, and other related explanations.

2.1. Literature contradicting that gypsum formation is expansive

Hansen [6] described work on sulfate attack conducted by Thorvaldson et al. during the early part of this century. Thorvaldson et al. used mortar-bar specimens made of one part C_3S + five parts sand and others made of one part C_2S + five parts sand. In 2 and 8% solutions of Na_2SO_4 , these specimens expanded no more than those stored in water. The 1:5 C_2S mortar expanded less than 0.01% after 22 years exposure in 2% Na_2SO_4 solution. However, in 8% Na_2SO_4 solution the mortar began to expand slowly after 3 years and reached an expansion of 0.9% at the end of 22 years. This expansion was attributed to a slow alkali-silica reaction on the quartz sand. In another test described by Lea [7], Thorvaldson et al. found that the 1:10 mortar bars of C_3S expanded 0.5% after 12 years of exposure in 2.1% Na_2SO_4 solution.

Hansen [6,8] presented a possible scenario about the role of gypsum formation during sulfate attack. He pointed out that if gypsum formation is expansive, the source of expansion should be due to the volume increase during the reaction of CH with sulfate ions to form gypsum. He did not believe that the gypsum generated by through-solution mechanism can form solid reaction products in a capillary cavity to occupy a larger volume than that of the cavity plus the volume of the solid CH that dissolves and takes part in the reaction. If the gypsum formed in a capillary cavity oc-

cupies a larger volume than that of the cavity plus the volume of the solid CH consumed in the reaction, it is only possible when gypsum crystals form in situ or on surfaces of CH particles (i.e., by topochemical reaction mechanism). He also believed that the precipitation of gypsum in the gel pores may not cause swelling of the pores by absorbing water, since the surface of the gypsum formed may not be appreciably greater than that lost by the dissolution of CH crystals. He noted that if there is a force exerted, it is due to hydraulic pressure but not due to the crystal growth.

Mather [9] does not believe that gypsum formation is always destructive. He did not agree that gypsum formation during sulfate attack on cement paste causes expansion. He supported Hansen's theory and pointed out that gypsum formation results primarily from the reaction of sulfate ions and calcium ions by a through-solution mechanism. Gypsum forms from the supersaturated solution by evaporation and subsequent precipitation. During this process, no expansion would be expected. He used an analogy to support his argument: "You cannot break a bottle full of saturated solution by removing the cork and letting the water evaporate."

2.2. Literature supporting that gypsum formation is expansive

Mehta [10] stated that the gypsum formation causes expansion and spalling based on his experimental study on alite paste. However, in his testing [11] alite mortar prisms were exposed to a 10% sulfate solution (5% Na_2SO_4 + 5% $MgSO_4$) and only short-term (75 days) expansion data were provided. The data showed no obvious expansion. The long-term results displayed that the loss of adhesion and strength was more evident than expansion.

Nielsen [12] conducted microscopic examination on thin sections of cement paste exposed to each of 0.07 M Na_2SO_4 , $MgSO_4$, and $FeSO_4$ solutions for 2 months. He gave no information on the C_3A content of the cement. He found that gypsum was the predominant reaction product while ettringite was rarely formed in an amount that was sufficient to be reasonably considered as a source for the formation of cracks. In Na_2SO_4 solution, the ettringite crystals found in cracks formed after occurrence of the cracks.

Bonen and Cohen [13,14] conducted microstructural analyses on Portland cement paste exposed to magnesium sulfate solution for 1 year. They observed the formation of a "surface double-layer" that was composed of brucite and gypsum layers and a sequential formation of inner layers of gypsum, similar to the occurrence of lise gang bands. Ettringite and monosulfate were only found in minor amounts.

Bonen and Sarkar [15] studied the replacement of the CH by gypsum in the interfacial zone (a zone between aggregates and bulk paste) along the boundaries of the aggregate particles. They found that thick deposits of gypsum up to 50 μm wide precipitate by a through-solution mechanism. The crystallization pressure of gypsum produced tensile stresses

and caused disruptive expansion. Their conclusion contradicted Hansen's paper [6] suggesting that gypsum forming by a through-solution mechanism cannot cause expansion.

Yang et al. [16] also studied the process of sulfate attack and the role of the interfacial zone during the attack process. Their conclusion is similar to that of Bonen and Sarkar—that sulfate reacts with CH and AFm in the interfacial zone, leading to expansion and cracking.

Wang [17] reported the results of X-ray diffraction (XRD) examination of the solid phases present at different depths in Portland cement paste attacked by Na_2SO_4 solution. He considered that more physical damage was caused by the formation of gypsum than by the formation of ettringite.

Gonzalez and Irassar [3] investigated the sulfate attack mechanism on four cements with low- C_3A content (three cements containing 0% C_3A , one cement containing 1% C_3A). The C_3S content in these cements varied from 40 to 74%. Their data showed greater expansion for the cement mortar specimens with higher C_3S content. The mortar made with 74%- C_3S cement reached an expansion of 0.112% after 180 days of exposure. Their XRD analysis showed that gypsum had formed in those specimens after 90 days of sulfate immersion. These gypsum peaks may suggest the correlation between expansion of the specimens and gypsum formation. However, they still attributed the expansion to the formation of ettringite, which was only observed by XRD in those specimens after 360 days of sulfate exposure. They concluded that "The great expansion was measured for high C_3S -content cement and the expansive formation of ettringite in this case was attributable to localized gypsum formation."

Ping and Beaudoin [18,19] suggested a theory based on the principles of chemical thermodynamics. They pointed out that the expansive force resulted from crystallization pressure. There are two conditions for the occurrence of crystallization pressure: (1) the solid product should form and grow in a confined space; (2) activity product of reactants in the pore solution should be greater than the solubility product of the solid products under atmospheric pressure. Theoretically, any solid product—not only ettringite—may produce crystallization pressure and cause expansion if the above two conditions are met. They suggested that in theory gypsum formation can be one of the principal causes of expansion during sulfate attack.

2.3. Other related explanations

Mehta [20] provided an explanation of the nature of deterioration of gypsum formation due to sulfate attack. He sug-

gested that due to gypsum formation and sulfate adsorption by CSH, the reduction in stiffness of concrete is preceded by large expansion due to ettringite formation.

Gollop and Taylor [21] conducted a series of tests dealing with sulfate attack on Portland cement paste. Using both microstructural and microanalytical techniques, it was determined that two zones of attack can form following a sequence of reactions. One zone was predominantly composed of ettringite and the other was composed of gypsum. Although no expansion measurement was reported, the microstructural features indicated that cracking was limited to the zone of gypsum formation. Their report tended to exclude the generally accepted idea that expansion is a direct and immediate result of ettringite formation. It was also suggested that gypsum formation is not responsible for the expansion. The most likely reason for the cause of expansion as suggested is the absorption of water by the "cement gel." The type of the cement gel and how it caused expansion, however, were not detailed.

3. Experimental investigation

3.1. Materials and experimental procedures

To simplify the chemical reaction process and eliminate the effects of ettringite formation, the suitable materials for the research on the sulfate attack caused by gypsum formation are the cement minerals: C_3S or C_2S . In this study, two kinds of C_3S were used. A limited amount available as a gray powder alite was provided by Concrete Materials Laboratory at Purdue University, which consisted of monoclinic crystals with small amount of impurities such as MgO , Al_2O_3 , Fe_2O_3 , and alkalis. Another is a white powder C_3S provided by the Construction Technology Laboratory (CTL, Skokie, IL), which consisted of triclinic crystals without impurities. A commercial-grade bulk silica fume (SF) was used to investigate its role on gypsum formation and the ensuing consequence on the mechanism. The chemical compositions of the materials used as obtained by energy dispersive X-ray analysis (EDXA) are shown in Table 1.

The alite was used to make paste specimens. The C_3S was used to make sand-mortar specimens. Deionized water was used as the mixing water. The mixture proportion for the alite paste was alite:water = 1:0.48 by mass. The mixture proportion for the C_3S mortar was C_3S :sand:water = 1:2:0.48 by mass. The sand used for making mortar specimens was 20–30 quartz sand meeting ASTM C 778.

The paste and mortar specimens incorporating silica fume were made with a silica fume replacement level of

Table 1
Chemical compositions of materials (mass percent)

	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	K ₂ O	Na ₂ O	P ₂ O ₅	MnO ₂	Total
Alite	72.34	24.76	0.65	0.34	0.92	0.64	0.00	0.24	0.04	0.14	100
C_3S	76.62	22.61	0.13	0.42	0.00	0.00	0.00	0.18	0.00	0.18	100
SF	0.19	98.25	0.36	0.21	0.00	0.62	0.28	0.14	0.00	0.00	100

10% of the binder (alite or C_3S). To better disperse the silica fume in the binder and to avoid the possible alkali-silica reaction, silica fume and binder were preblended dry in a roller blender with ceramic balls as the mixing media for over 48 h.

The mixing of the specimens followed the ASTM C 305. Prisms (8 by 16 by 80 mm) with acrylic resin end pieces were used for expansion measurement. Plastic molds, which have been described by Cohen and Bentur [22] and Kerdegari [23], were used for casting the beams. The specimens were demolded after 1 day and then were cured at $20 \pm 1^\circ\text{C}$ in a saturated lime solution. After 14 days of curing they were divided into several categories and exposed to different attacking solutions.

Exposure solutions were made by dissolving reagent-grade chemicals in deionized water. The chemicals used were sodium sulfate (Na_2SO_4) and ammonium sulfate $[(\text{NH}_4)_2\text{SO}_4]$. The SO_4^{2-} concentration in the sulfate solutions was 33,800 ppm [5% Na_2SO_4 or 4.65% $(\text{NH}_4)_2\text{SO}_4$], which is the same as the solution condition of ASTM C 1012. The solutions and specimens were kept in plastic containers with ample space between specimens. The containers with the specimens were stored in a constant temperature ($20 \pm 1^\circ\text{C}$) room. The solutions were replenished periodically—once a week for the first month after the start of the exposure, and then once every two months.

3.2. Results and discussion

3.2.1. Expansion of alite paste specimens, with/without silica fume, in Na_2SO_4 solution

The first experiment conducted was the testing of the expansion of alite paste specimens, with and without silica fume, exposed to Na_2SO_4 solution. The results are shown in Fig. 1.

In 5% Na_2SO_4 solution, alite paste specimens showed no expansion for a period of approximately 360 days. After this “dormant” period the specimens began to show significant expansion. The expansion reached 0.10% at 480 days. Alite/silica fume paste specimens showed no expansion. This behavior was similar to the control alite paste specimens immersed in saturated limewater solution.

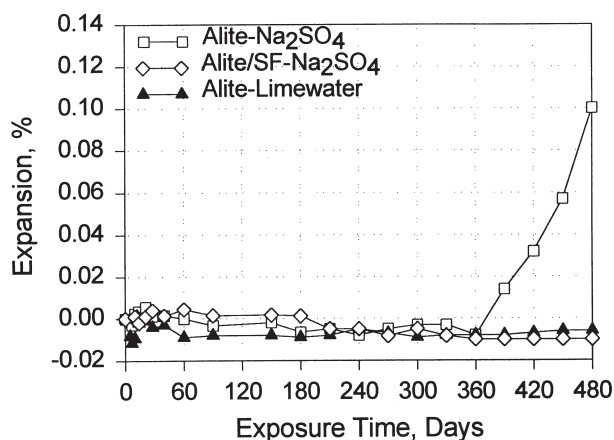


Fig. 1. The expansion of alite with/without SF paste in Na_2SO_4 solution.

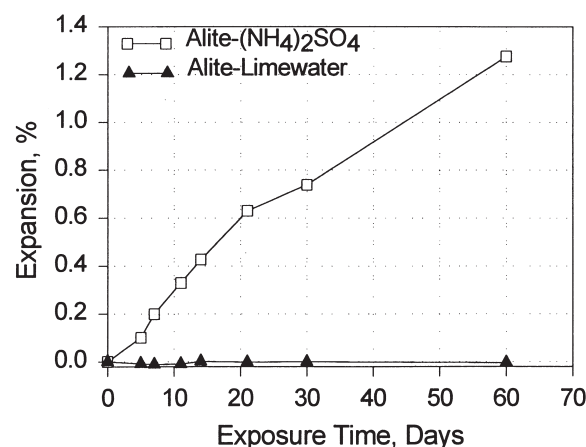


Fig. 2. The expansion of alite paste in $(\text{NH}_4)_2\text{SO}_4$ solution.

3.2.2. Expansion of alite paste specimens in $(\text{NH}_4)_2\text{SO}_4$ solution

$(\text{NH}_4)_2\text{SO}_4$ is a chemical that can attack Portland cement concrete severely and rapidly [24,25]. The cause is generally attributed to the reaction between the sulfate ions and C_3A that produces ettringite. The ammonium sulfate may also attack the CSH. Fig. 2 indicates that the attack of $(\text{NH}_4)_2\text{SO}_4$ solution on alite paste specimens was severe considering that there was no C_3A , and thus no ettringite formation. It also shows that expansion increased rapidly. After only 60 days of exposure, the specimens expanded to an average strain of 1.28%.

3.2.3. Expansion of C_3S mortar specimens, with/without silica fume, in Na_2SO_4 solution

To further study the behavior of C_3S hydration products under sulfate attack, another experiment was carried out specifically to study the behavior of the C_3S mortar specimens, with and without silica fume, when exposed to Na_2SO_4 solution. The results are shown in Fig. 3. In Na_2SO_4 solution, the C_3S mortar specimens showed no expansion until about 40 days of exposure. This dormant period of 40

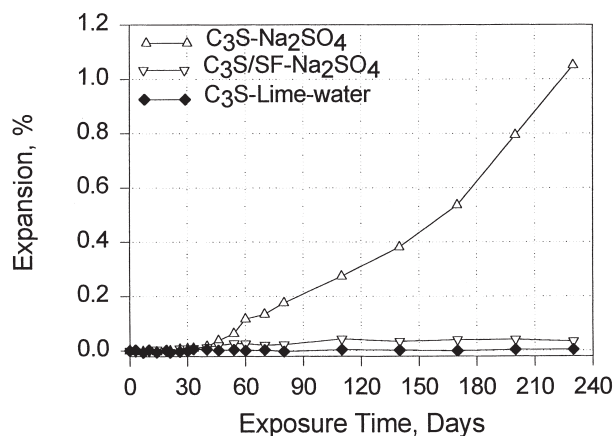


Fig. 3. The expansion of C_3S with/without SF mortar in Na_2SO_4 solution.

days was shorter than that for alite paste (360 days) (Fig. 1). After 40 days (Fig. 3), the C_3S mortar specimens expanded rapidly at a rate faster than that of alite paste specimens. At 230 days of exposure, the expansion reached 1.05%. However, the specimen with silica fume expanded to about 0.04%, which is only slightly greater than that in the control limewater solution.

3.2.4. Physical appearance

The physical appearance of the specimens after different periods of the exposure are shown in Fig. 4. For alite paste specimens in Na_2SO_4 solution (expansion shown in Fig. 1), there were cracks that formed along the edges of the specimens (Fig. 4). The surfaces were scaled. In alite/silica fume paste specimens there was no visible damage. For alite paste specimens in $(NH_4)_2SO_4$ solution (expansion shown in Fig. 2), there were cracks formed on the surfaces and along the edges (Fig. 4). The specimens were severely bent due to the uneven fast expansion. For C_3S mortar specimens in Na_2SO_4 solution (expansion shown in Fig. 3), there were cracks formed around the sand aggregates (Fig. 4). Similar to alite/silica fume paste specimens, there was no damage on C_3S /silica fume-mortar specimens (Fig. 4). The physical appearance appeared to be consistent with the expansion results. Those specimens with high expansion showed cracks and other damage, while those with little or no expansion showed no physical damage.

In the case of specimens with high expansion [i.e., alite paste in Na_2SO_4 solution and in $(NH_4)_2SO_4$ solution, C_3S mortar in Na_2SO_4 solution], the acrylic end pieces on the specimens were cracked (as marked by the arrows in Fig. 5) due to tensile forces from the specimens, while those acrylic end pieces on the specimens with silica fume showed no expansion (i.e., alite with silica fume paste in Na_2SO_4 solution and C_3S with silica fume mortar in Na_2SO_4 solution) did not show any cracks.

3.2.5. XRD examination

The solid phases in the specimens after different exposure periods were examined using XRD. The XRD patterns of alite paste and C_3S mortar specimens, with and without silica fume, are shown in Fig. 6.

As expected, the main product formed in all specimens was gypsum; the amount of gypsum was greater in the specimens without silica fume. Some unhydrated C_3S was found to be present in all the specimens.

After being exposed to Na_2SO_4 solution for 440 days, alite paste specimens showed high gypsum peaks in its X-ray pattern, while alite with SF paste showed small peaks of gypsum.

For alite paste specimens exposed to $(NH_4)_2SO_4$ solution, there was a large amount of gypsum formed even though the exposure time was only 60 days. This could be related to the large expansion observed. The calcium hydroxide (CH) peaks

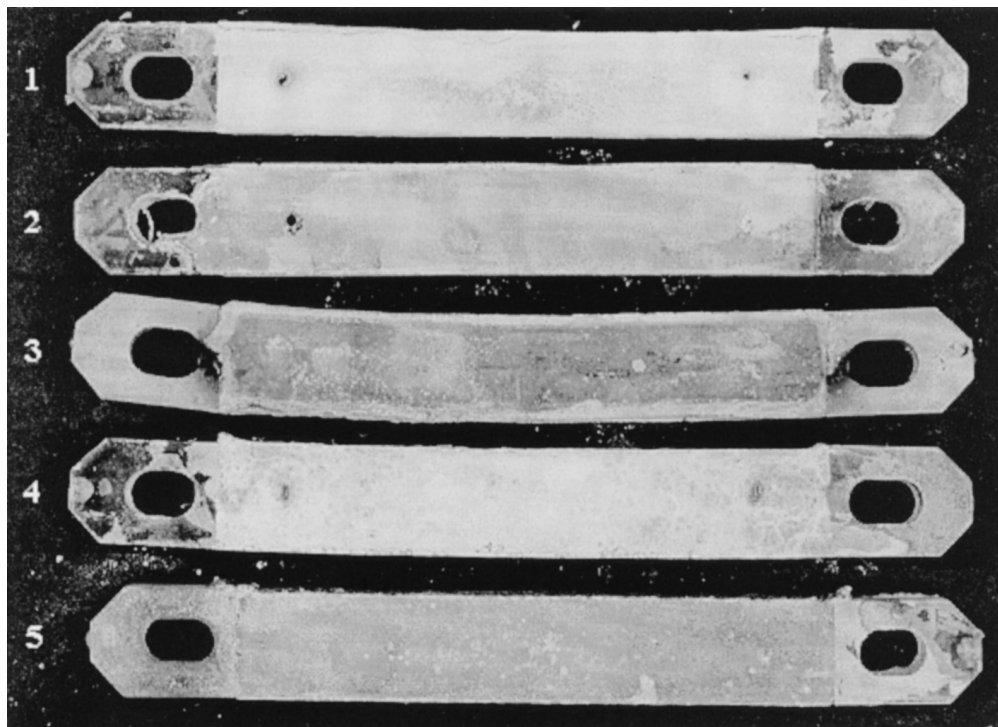


Fig. 4. The physical appearance of the specimens after different exposure period: (1) alite paste in Na_2SO_4 solution for 440 days; (2) alite/SF paste in Na_2SO_4 solution for 440 days; (3) alite paste in $(NH_4)_2SO_4$ solution for 60 days; (4) C_3S mortar in Na_2SO_4 solution for 220 days; (5) C_3S /SF mortar in Na_2SO_4 solution for 220 days.

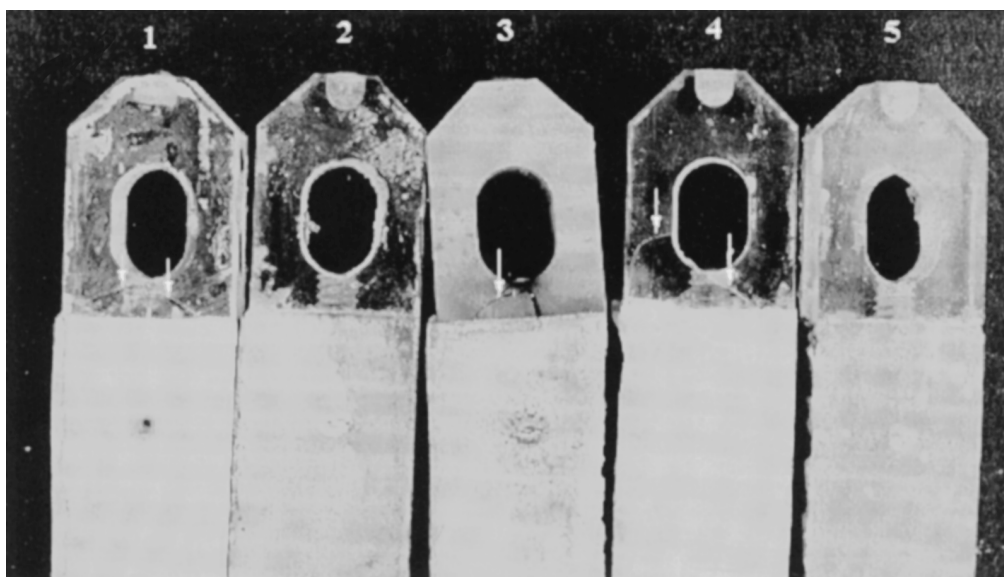


Fig. 5. Cracks formed on the acrylic end pieces of the specimens (marked by arrows).

were also high. The large amount of CH in the specimens may transform to gypsum while being exposed to sulfate solution.

For C_3S mortar specimens exposed to Na_2SO_4 solution for 220 days, the observation was similar to that of alite paste specimens. More gypsum was formed in C_3S mortar without SF than in the mortar with SF, even considering the

dilution effect. Large quartz peaks are evident in the XRD patterns due to the sand in the specimens.

4. Summary

In this paper research results are reported, which suggest that gypsum formation during sulfate attack is expansive.

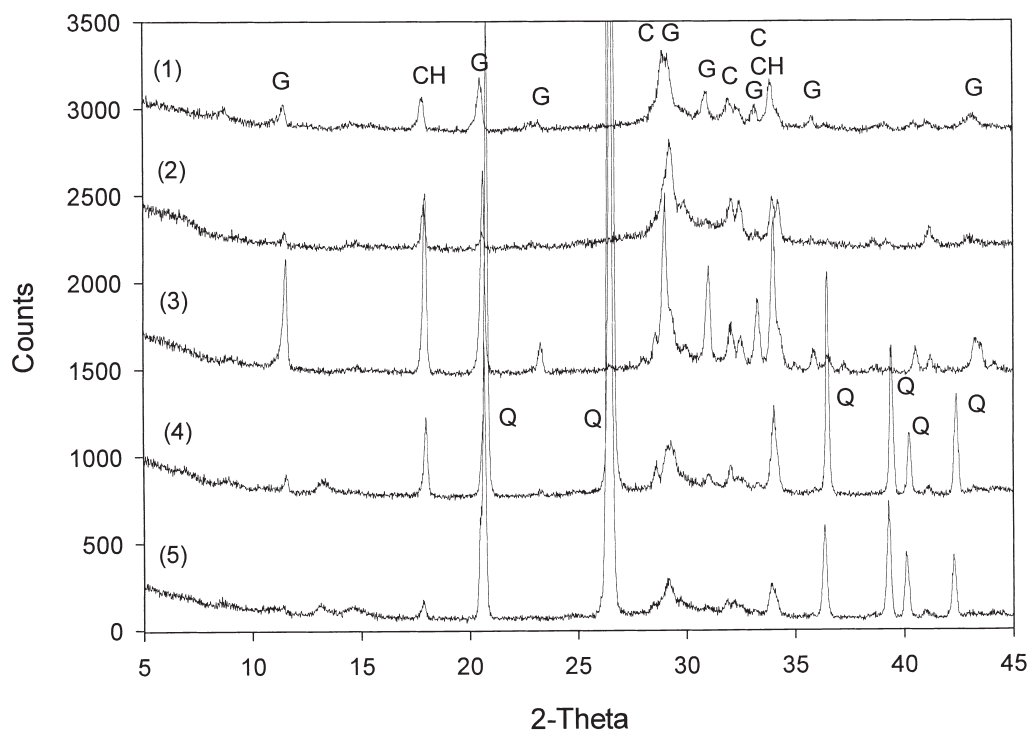


Fig. 6. The XRD patterns of the specimens (G, gypsum; C, C_3S ; CH, $Ca(OH)_2$; Q, quartz): (1) alite paste in Na_2SO_4 solution for 440 days; (2) alite/SF paste in Na_2SO_4 solution for 440 days; (3) alite paste in $(NH_4)_2SO_4$ solution for 60 days; (4) C_3S mortar in Na_2SO_4 solution for 220 days; (5) C_3S /SF mortar in Na_2SO_4 solution for 220 days.

The exact mechanism (i.e., by topochemical reaction mechanism or by through-solution mechanism), however, is not clear. The results of these findings indicate that sulfate attack mechanism is complicated and the expansion and cracking of Portland cement concrete should probably not be exclusively attributed to ettringite formation. Tensile stresses during gypsum formation may also play a role in expansion and the subsequent cracking.

Acknowledgments

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