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Delayed ettringite formation in heat-cured Portland cement mortars

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Abstract

A Portland cement mortar was subjected to elevated temperature curing at 100°C for 12 h and then stored under water at room temperature. Expansions, attributable to delayed ettringite formation, were found to develop over a period of 1 year. Sulfate ions released to the pore fluid at elevated temperatures, and partly sorbed by C-S-H gel, evidently formed ettringite in the outer products and the paste–aggregate transition zones during subsequent water storage at room temperature. The results of X-ray microanalyses implied that a potential for ettringite band formation had been established in the mortar. Investigations of microstructural features by backscattered electron imaging indicated that the expansion was caused by generation and extension of these ettringite bands. No evidence in support of an alternative mechanism based on a homogeneous expansion of the cement paste could be found.

Keywords: Ettringite; Heat curing; Mortar; Interfacial transition zone; Microstructure; X-ray microanalysis

The observed expansion of some laboratory and field Portland cement mortars and concretes, which were exposed to elevated temperatures and subsequently stored at room temperature in moist conditions, has been attributed to delayed ettringite formation (DEF) by many researchers [1–9]. Some authors [10,11], however, do not believe that the deterioration of the steam-cured field concretes can be caused by DEF alone without the aid of other factors, for example, alkali-silica reaction expansion (ASR), freeze-thaw cracks, or the presence of other microstructural flaws. In support of this view, it is generally recognised that expansions due to DEF occur much less readily in cement pastes, which are free from paste-aggregate bond defects, than in mortars or concretes. Expansions were reported by Odler and Chen [12] in one high sulfate and high aluminate Portland cement paste cured at 90°C, and by Lawrence [13] (and personal communication, 1995) in pastes made from three commercial cements (SO_3 = 3.2%, 3.3%, and 4.2%) and cured at either 80°C or 100°C. These observations showed that DEF acting alone might cause expansion in some commercial cement products cured at 80°C and above, but not in all [13,14]. Of course, when cracks are produced by ASR or by some other factor, ettringite may crystallize in these cracks and result in accelerated and enhanced expansions.

It is still difficult in practice to predict the occurrence of DEF expansion or to diagnose disruption of field concrete caused by DEF, because the mechanism of DEF expansion is not yet clearly established. According to investigations by optical microscopy and scanning electron microscopy (SEM), many ettringite bands can be observed in concretes or mortars (particularly in the transition zone between the paste and the aggregate) that have suffered from expansion induced by DEF. Discussion of the mechanism of DEF expansion in the literature concentrates on the interpretation of the relationship between expansion and delayed ettringite band formation. There are two principal hypotheses, the ettringite crystal growth pressure theory [1–9] and the uniform paste expansion theory [15–19].

The former hypothesis suggests that pressure from ettringite crystals growing in preformed microcracks in the transition zone between the cement paste and the aggregate widens these cracks and causes the observed expansion. This theory was disputed by Johansen et al. [15] and Taylor [18], who suggested that the degree of supersaturation of ettringite in the pore solution of the cementitious material was insufficient to generate sufficient crystal growth pressure to achieve this expansion. Diamond [9], however, suggested on the basis of the basic concepts of fracture mechanics that a modest degree of ettringite supersaturation is enough for ettringite growth to extend the cracks, because the stress concentration factor at the tip of a long crack in concrete is large as a result of its quasi brittle nature.

Alternatively, the uniform paste expansion theory suggests that expansion of the cement paste matrix occurs, leaving gaps around the aggregate grains. Subsequently,

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newly crystallising ettringite rapidly fills the cracks produced by this expansion to result in ettringite band formation. On this basis, therefore, ettringite band formation does not contribute directly to the observed expansion. Johansen et al. [16] and Lee [17] found that the widths of the ettringite bands around the aggregate were proportional to the size of the aggregate. They attributed this phenomenon to the effect of uniform expansion of the paste. Careful microscopic investigations by Diamond [9] and Lawrence [20], however, showed that many ettringite bands and rim cracks form as portions of a continuous crack network distributed throughout the concrete, which developed partially, rather than completely, around the aggregate grains. Some aggregate grains do not have ettringite or crack rims. These observations seem to be incompatible with the idea of homogeneous cement paste expansion. The results of X-ray microanalysis by Lewis et al. [21] and Lewis [22] suggested that portions of sulfate and aluminate species may be intimately mixed with C-S-H immediately after the heat cure due to dissolution and decomposition of ettringite. The sulfate content in the C-S-H gel decreased, while ettringite recrystallised during the period of expansion. At the end of the expansion process, the gel was only intermixed with AFm. Lewis et al. [21] postulated that recrystallisation of ettringite initially occurred in C-S-H gel and then in cracks and voids. Ettringite recrystallisation in the C-S-H gel might have caused the uniform expansion of the paste.

Both hypotheses seem to lack conclusive evidence and cannot be regarded as established. In the current work, a set of laboratory experiments was designed to investigate the expansion caused by DEF, carefully avoiding the effects of ASR and inadequate precuring. To reduce the induction period of DEF expansion, heat curing at 100°C was used in this investigation. On the basis of previous investigations reported in the literature [1,13,18,20], the increase of heat curing temperature, such as from 80°C to 100°C, may reduce the induction period of expansion and increase the ultimate expansion. Lawrence [13] was able to show gradually increasing expansive behaviour as the initial curing temperature was increased. In the present experiments, the process of ettringite band development was observed, as were variations in the ettringite content and C-S-H gel composition of the heat-cured mortars.

1. Experimental

The chemical and mineralogical compositions of the U.K. Portland cement used in this research are listed in Table 1. British standard siliceous sand was used to make the mortars. The dominant phase was quartz. Although ASR cannot be absolutely ruled out for these specimens, no evidence for formation of alkali-silica gel could be observed by backscattered electron (BSE) imaging in either the mortars cured at 100°C and subsequently stored at room temperature under water, or those continuously cured at room temperature, for up to 1 year. The specimens were stored under water, suggesting that most of the alkali was rapidly removed, again making ASR an unlikely cause for expansion.

A water-to-cement ratio of 0.5 and a cement-to-sand ratio of 0.33 were used to cast 40 × 40 × 160-mm mortar prisms. The cast prisms, while still in their steel moulds, were precured at room temperature in a moist environment for 2 h and then heated at a rate of 20°C/h to 100°C. They then were cured at 100°C for 12 h, cooled to room temperature at a rate of 10°C/h, demoulded, and labelled WM100, and stored under water for up to 1 year after demoulding at 1 day. The control mortar specimens, labelled WMRT, were continuously cured at room temperature (about 20°C) under water for 1 year. The lengths of the mortar prisms were measured at regular intervals after demoulding using a length comparator.

The Camscan series 2 SEM was equipped with a BSE detector and an energy dispersive X-ray spectrometer (EDS). All microstructural features of the mortars were observed by BSE imaging in polished section. Chemical composition of gel-like phases in the cement paste matrix of the mortars was determined by EDS using the ZAF correction procedure. The specimens for microstructural examinations were cut directly from the mortar prisms.

The amount of ettringite that formed in the cement matrix in the mortars was determined quantitatively by means of a Philips X-ray diffractometer. The X-ray diffraction (XRD) pattern of the sample was matched to those of the primary standards in the scanning range from 7 to 30 degrees (2ϑ) by a least-squares procedure. The diffraction profiles of the pure ettringite phase and the sands were stored in a computer as primary standards. Most of the sand component of the mortar was separated by grinding and sieving, but this left a residual fraction intimately associated with the cement paste. The weight fraction of ettringite and the residual fraction of sand in the paste were determined by quantitative X-ray diffraction analysis (QXDA). The sand fraction was deducted from the analysis total.

Table 1
Chemical and mineralogical composition of the cement

Oxides	Wt. (%)	Phases	B-cal (%)	MB-cal
SiO_2	20.7	Alite	59.2	73.6
Al_2O_3	4.9	Belite	14.9	4.9
Fe_2O_3	2.8	C_3A	8.3	8.1
CaO	63.9	Ferrite	8.4	7.9
MgO	2.2			
SO_3	3.2			
K_2O	0.7			
Na_2O	0.1			
Na_2O eq	0.56			
Total	98.5	Total	90.8	94.5

B-cal: mineralogical composition calculated by Bogue method; MB-cal: mineralogical composition calculated by the modified Bogue method [23]; Na_2O eq: equivalent Na_2O.

2. Results and discussion

2.1. Expansion

Expansion of the mortars heated at 100°C for 12 h was observed to start within 90 days and was complete after about 1 year (Fig. 1). The control mortars showed no significant expansion.

2.2. XRD examination

As shown in Fig. 2, most of the ettringite in the mortars cured at room temperature formed at the initial stage of hydration (within 7 days). Ettringite could not be detected in the mortar samples immediately after heat curing. The overall amount of ettringite in the heat-cured mortar started to increase after 7 days of water storage at room temperature and then continued to increase. It was, however, generally lower than that of the mortar cured at room temperature.

The XRD patterns of the 100°C cured mortar and the control mortar are shown in Fig. 3. Hydrogarnet was detected, by the presence of four XRD peaks, in the heat-cured mortars throughout the period of water storage at room temperature, but monosulfate could not be detected. In contrast, monosulfate appeared in the room-temperature–cured control mortars after 7 days of hydration, but hydrogarnet was not detected even after hydration for 1 year.

2.3. Microstructures of mortars cured at room temperature

Microstructures of the mortar continuously cured at room temperature for 1 year are shown in Fig. 4. Only empty cracks could be observed (Fig. 4a), at least some of which were produced on drying during sample preparation for SEM. Some regions of high monosulfate concentration appear in the mortar (Fig. 4b), whereas few ettringite-concentrated areas could be found in the BSE images.

2.4. Development of ettringite bands in mortars cured at 100°C

In the mortar examined immediately after the heat cure, no concentration of ettringite could be found by BSE imaging, and no significant differences were observed in the frequency or the width of cracks or gaps in the transition zone between the cement paste and the aggregate grains compared with the mortars hydrated only at room temperature (Fig. 5).

Indications of initial ettringite band formation and areas of ettringite concentration could be identified in the heat-cured mortar subsequently stored under water at room temperature for 90 days by BSE imaging and EDS, respectively. These bands developed both in the transition zone between the cement paste and the aggregate (Fig. 6a) and in the cement–paste matrix (Fig. 6b). Most of the bands started to form in the region near the surface of the prism. At this initial stage of ettringite band formation, these bands, as shown in Figs. 6a and 6b, seemed to have formed directly from gel-like hydration products rather than having been deposited in preformed cracks as passive fillings (Fig. 6a). Furthermore, the empty cracks linking the ettringite bands were distinctly narrower than the ettringite bands.

In the heat-cured mortars after 155 days of water storage at room temperature, ettringite bands had clearly developed, both in the aggregate–paste transition zone and in the cement–paste matrix, in the region near and approximately parallel to the surface of the prisms. More ettringite bands could be observed in the transition zone than in the paste matrix. These bands passed through the transition zones along one or two sides of the aggregate rather than completely around the aggregate (Fig. 7).

At later stages of the prism expansion, ettringite bands had developed from the surface region to the central area of the prism. Many large aggregate grains were covered by ettringite bands from one, two, or occasionally three sides

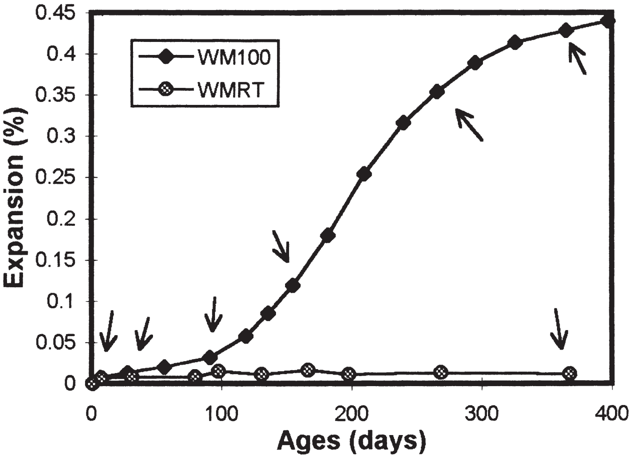

Fig. 1. Expansions of the heat-cured and the control mortars. The mortar prisms were cut at the ages indicated by arrows for microstructural observations.

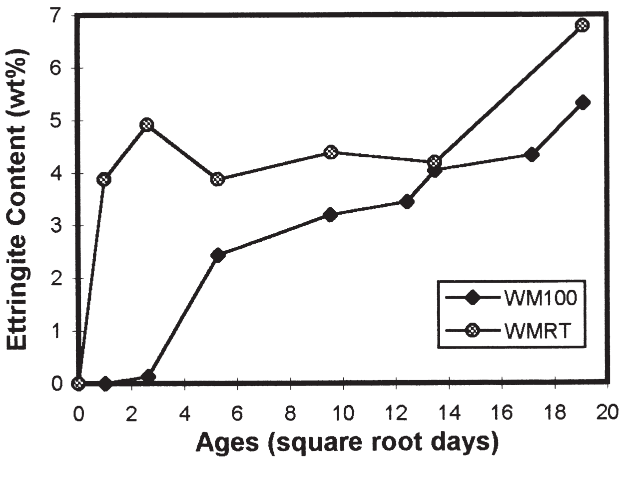

Fig. 2. Ettringite contents of the cement pastes in the heat-cured mortars (WM100) and in the control mortars (WMRT) determined by QXDA.

(Fig. 8a). Some ettringite bands were observed to have developed in the paste matrix and formed bridges between the aggregate grains (Fig. 8b).

The distinct microstructural characteristics of the cement paste in the expanded, heat-cured mortars were similar to those of the heat-cured cement paste reported previously [24], which showed large expansions after long-term water storage at room temperature. The “two-tone” inner products could be clearly identified in the heat-cured mortars after 90 days of storage (Fig. 9a). The outer and lighter zone was formed during the period of heat curing, whereas the inner and darker zone was formed during subsequent water storage at room temperature following the heat cure. These inner zones had dense and uniform microstructural features at the early stages of the expansion process. Empty cracks free from ettringite appeared in some of them during the later stages of the expansion process. They seemed to appear preferentially in the inner products free from the presence of any remaining anhydrous alite (Fig. 9b). Most of these empty cracks were isolated in the inner products and were not connected with the main network of cracks or ettringite bands that developed throughout the whole mortar. Although such cracks appeared in some of the inner products, they did not appear in many of them (Fig. 8b), so that it seemed unlikely that they resulted from homogeneous cement paste expansion. Areas of ettringite concentration could be observed in a few of the inner products (arrowed in Fig. 9b). In a similar manner to those present in the outer products, some of them could probably develop into ettringite bands.

The uniform paste expansion hypothesis predicts that empty gaps should be formed around the aggregate grains and subsequently filled by ettringite, but very few such gaps were observed in the current mortars examined at any stage of the expansion process. Instead, the cracks observed were always either completely empty or well filled by ettringite (i.e., ettringite bands) from the beginning to the end of expansion. Very few aggregate grains were fully surrounded by ettringite bands, but such bands did appear in the paste–aggregate transition zone and simultaneously in the paste matrix from the early stages of expansion (Figs. 6b and 7). If the uniform paste expansion is operating in the system, why is the paste itself cracked at a very early stage of expansion? Therefore, it is concluded that no evidence was obtained to support the paste expansion hypothesis.

Furthermore, the following microstructural features of the ettringite bands seem to be explicable only in terms of the crystal growth theory. As shown in Fig. 10a, some ettringite bands formed from areas of ettringite concentration in the cement–paste matrix and subsequently developed into the transition zone. It is a very common microstructural feature in the mortars suffering from DEF expansion that ettringite bands of different lengths formed between the aggregate and the portlandite rim. Portlandite deposits inti-

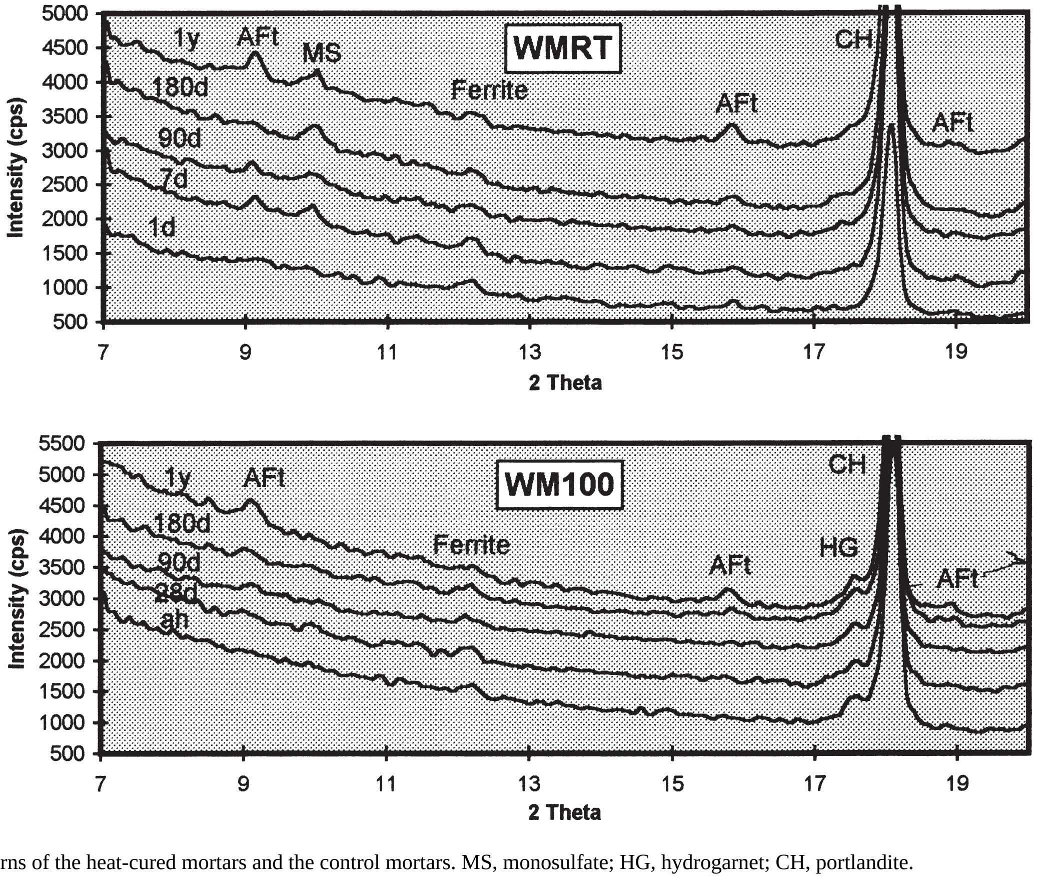

Fig. 3. XRD patterns of the heat-cured mortars and the control mortars. MS, monosulfate; HG, hydrogarnet; CH, portlandite.

mately on the surface of the aggregate grains in nonexpansive mortars, such as mortar immediately after the heat cure and the control mortar (Figs. 4 and 5). Fig. 10b shows a typical feature of such an ettringite band in development and suggests a process in which the ettringite band develops around the grain of aggregate. It can be seen that there is clear evidence for formation of an ettringite band between the aggregate and the calcium hydroxide rim. The space in the interfacial zone currently occupied by ettringite is not a former air bubble, because it is not spherical in shape. It is not space previously filled by water, because this space is so narrow that there is no reason why portlandite should form only around it rather than in it. The microstructural features of the crack developing around the aggregate grain and its surrounding paste are hard to associate with homogeneous or heterogeneous paste expansion. In addition, the portlandite band covering the ettringite band looks like a part of the original band developed directly on the surface of the same aggregate particle. They have a similar thickness and are continuously connected to each other. It seems that the calcium hydroxide rim covering the ettringite band was origi-

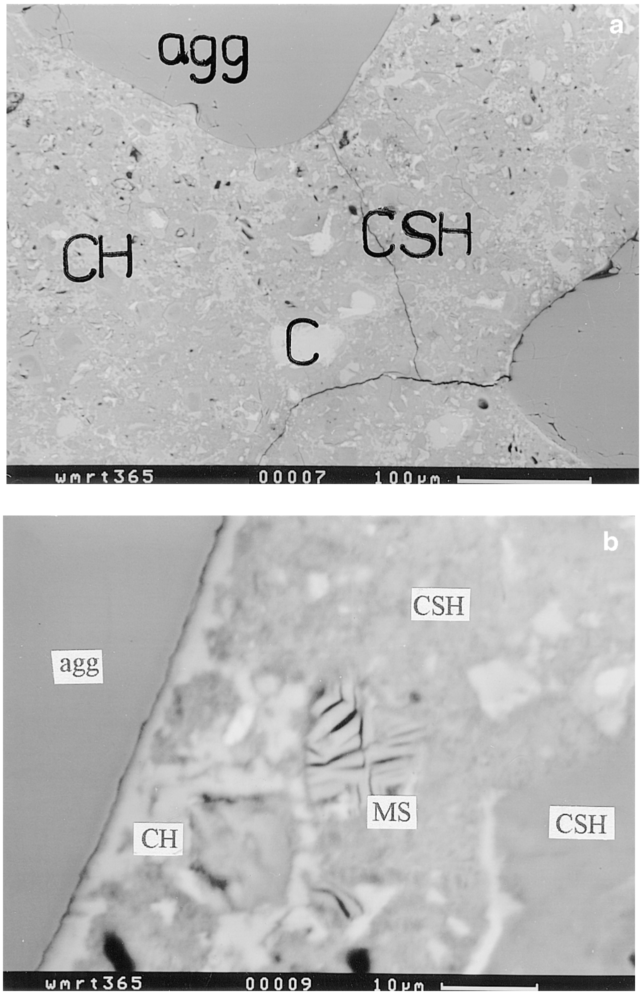

Fig. 4. (a, b) Microstructures of the control mortar cured at room temperature for 1 year. C, anhydrous clinker; CH, portlandite; agg, aggregate; CSH, C-S-H gel; MS, monosulfate.

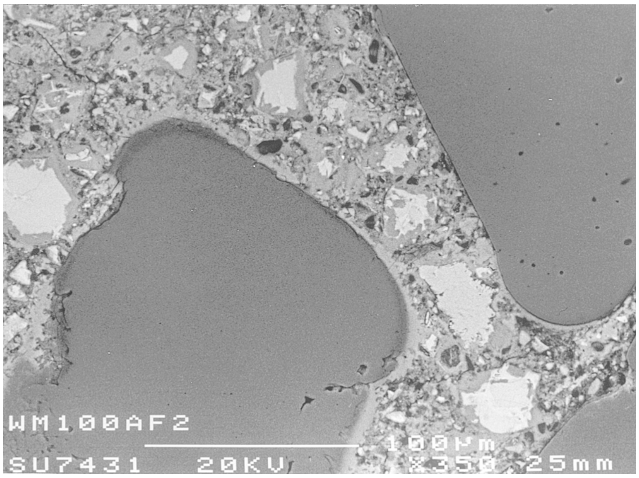

Fig. 5. Microstructure of the mortar immediately after heat curing.

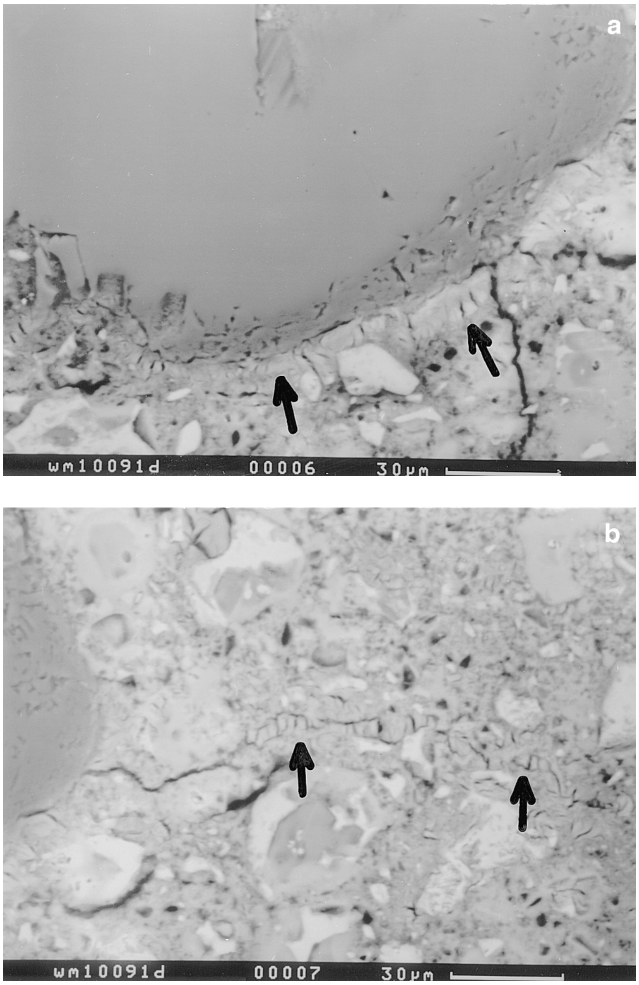

Fig. 6. (a, b) Initial formation of the ettringite bands in the heat-cured mortar after 90 days of storage.

nally in contact with the surface of the aggregate. The ettringite band subsequently developed in the transition zone between the aggregate and the CH rim, where the cohesion was weak, with the result that it pushed the calcium hydroxide rim away from the surface of the aggregate.

In the expansive heat-cured mortar, the width of the ettringite bands did not correlate with the size of aggregate grains they covered. Ettringite bands of similar width (5 to 10 μm) were observed to develop around the aggregate grains of different size and in the paste matrix.

2.5. *X-ray microanalysis of the mortars cured at room temperature*

X-ray microanalyses were carried out on the inner hydration products, i.e., C-S-H gel formed within the original boundary of the alite grain (ih), the outer hydration products, i.e., C-S-H gel formed outside the inner products (oh), and the areas of sulfoaluminate concentration, i.e., AFt and

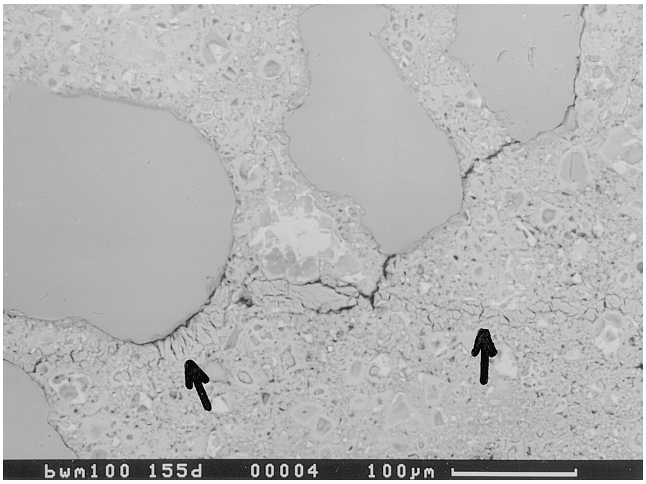

Fig. 7. Development of ettringite in the heat-cured mortar after 155 days of storage.

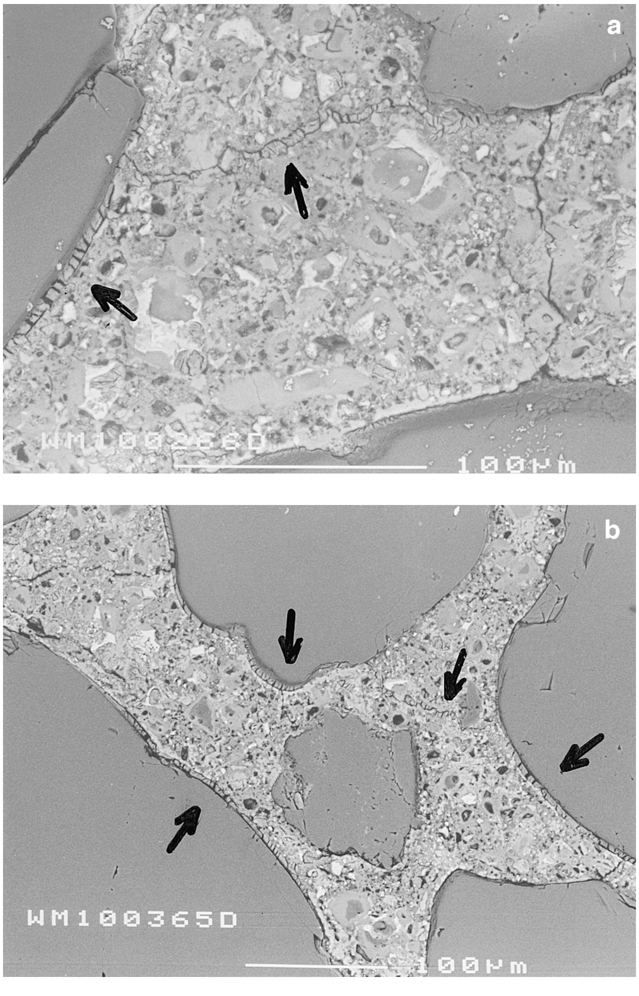

Fig. 8. (a, b) Ettringite bands in the heat-cured mortar after storage for (a) 266 days and (b) 365 days.

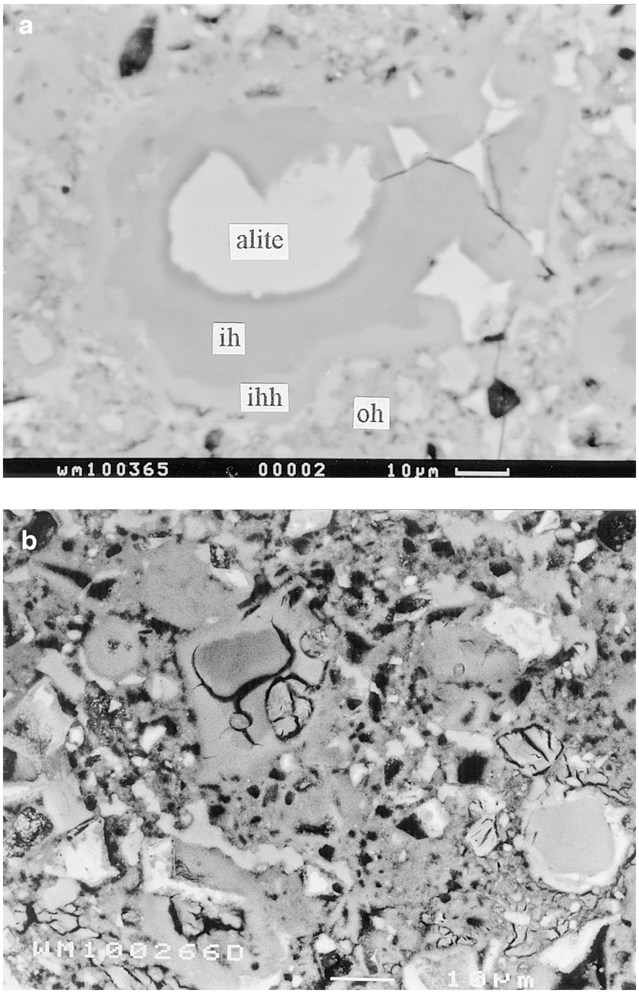

Fig. 9. (a, b) Microstructural characteristics of the cement paste in the mortar cured at high temperature and subsequently stored at room temperature for more than 266 days. ih, inner hydration products formed during the storage at room temperature after the heat cure; ihh, inner hydration products formed during the heat cure; oh, outer hydration products.

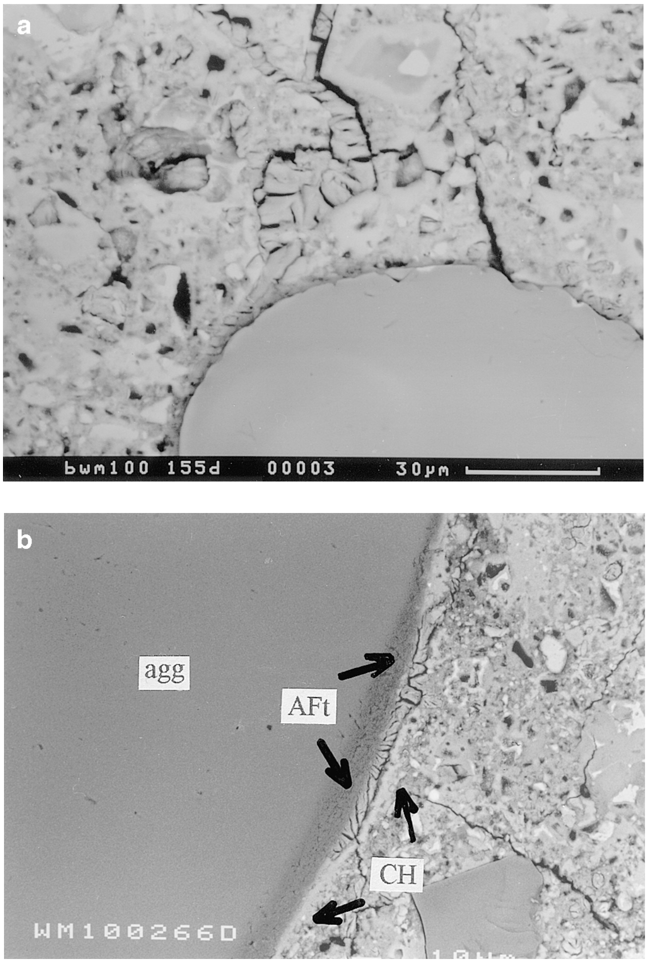

Fig. 10. Processes of ettringite band development in the heat-cured mortars stored for (a) 155 days and (b) 266 days.

AFm (sa), respectively. The results, shown in Figs. 11a and 11b, indicate that the dominant hydrate was monosulfate rather than ettringite in the areas of sulfoaluminate concentration (sa) in both the control mortars cured for 28 days (Fig. 11a) and for 1 year (Fig. 11b). The inner products (ih) had a significantly lower S/Ca ratio than the outer products, so that the analyses of the inner products are distinct from those of the outer products. Most of the ettringite in the control mortars was present in the outer products (oh), intermixed with C-S-H, AFm, etc. The S/Ca ratio in the outer products was lower for the mortar after storage for 1 year compared with that stored for 28 days, which suggested an increase of AFm in the outer products.

2.6. X-ray microanalysis in the heat-cured mortars

Separate X-ray microanalyses were performed in the outer and lighter zone of the inner products (ihh), formed during the heat cure, and in the inner darker zone (ih) formed during subsequent storage at room temprature (Fig. 9a). Few areas of sulfo-aluminate concentration could be found in the mortars immediately after heat curing. As shown in Fig. 12a, the inner products that formed during the period of heat curing contained higher sulfate contents than those in the control mortars. Most of the analyses of the outer products had S and Al levels similar to those of the inner products. These results, in conjunction with XRD analyses, provide further evidence that the sulfate ions are sorbed by the C-S-H gel during the period of heat curing, as suggested originally by Scrivener and Taylor [19] and Lewis et al. [21]. The Al/Ca ratio of the inner products in the mortar immediately after heat curing did not seem to be distinctly different from that in the control mortar.

After 90 days of water storage at room temperature, the heat-cured mortar showed a reduction in S/Ca and Al/Ca ratios for the inner products formed during the heat cure, and the outer products also showed reductions in these ratios. This indicates that the sulfate ions sorbed in the C-S-H gel tend to be expelled from the C-S-H gel (Fig. 12b). Much of the expelled sulfate subsequently reacted with aluminate to form ettringite. In contrast to the case of the control mortar, many analyses of the outer products overlapped with those

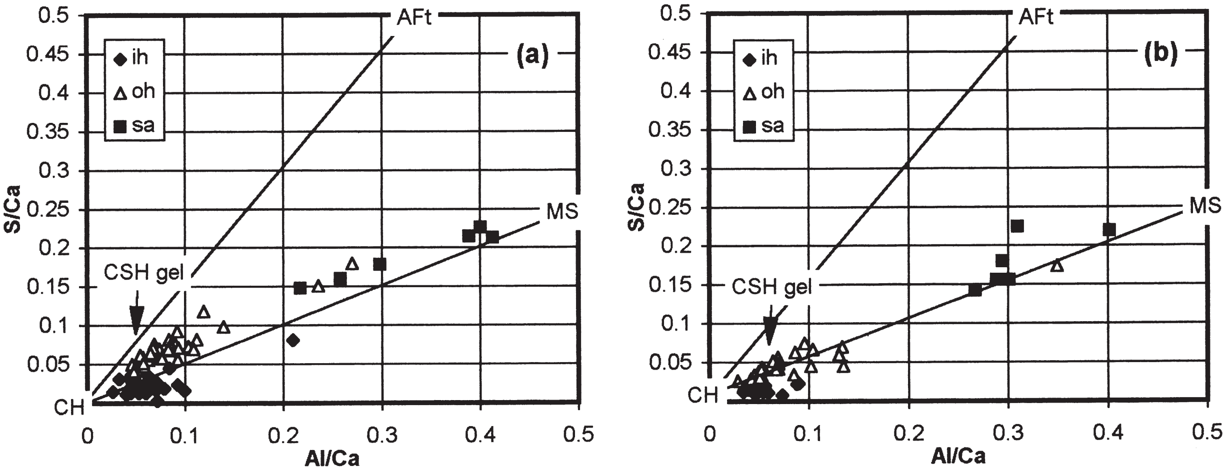

Fig. 11. Atomic ratio plots of S/Ca vs. Al/Ca of the control mortars cured for (a) 28 days and (b) 1 year. ih, inner products; oh, outer products; sa, sulfoaluminate hydrates; MS, monosulfate.

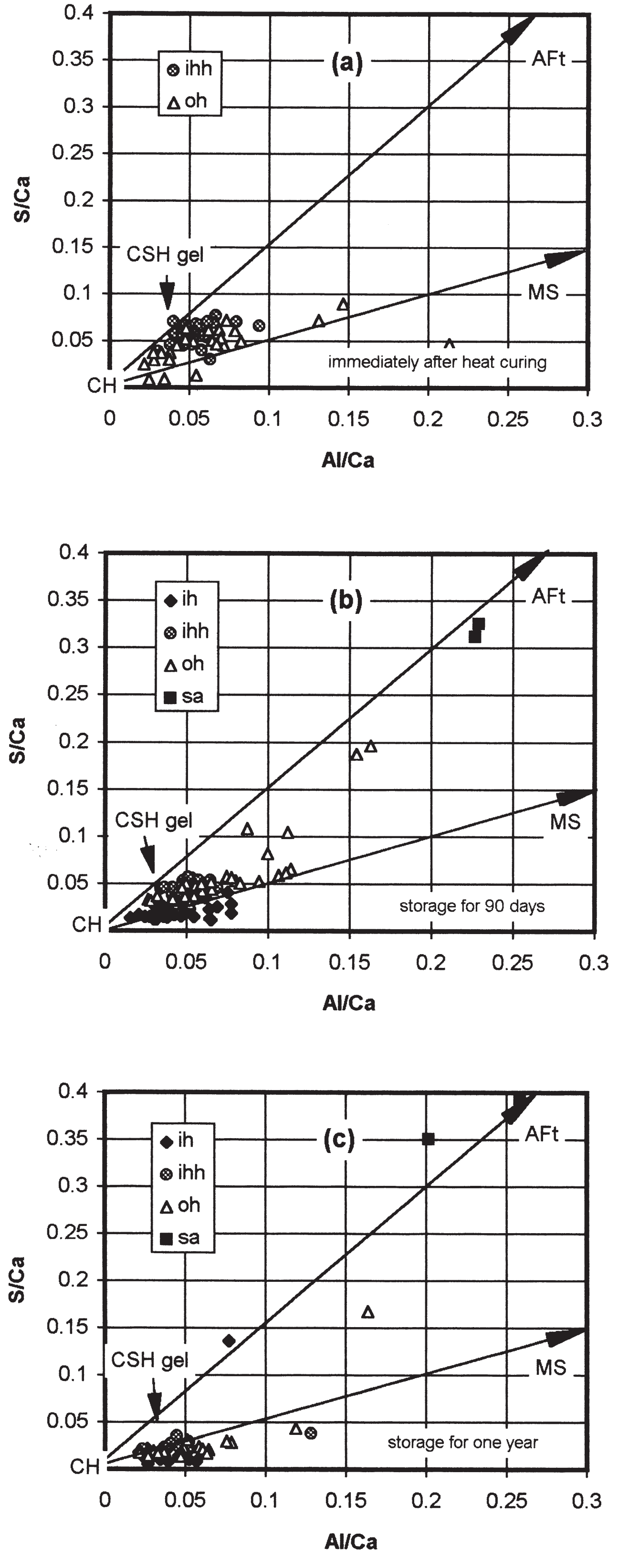

Fig. 12. (a–c) Atomic ratio plots of S/Ca vs. Al/Ca of the mortars cured at 100°C and subsequently stored at room temperature. ih, inner and darker zone; ihh, outer and lighter zone; oh, outer products; sa, sulfoaluminate hydrates; MS, monosulfate.

of the inner products formed during the heat cure, which implies that ettringite cannot intermix with these parts of the outer products. Hence, this ettringite formed bands or concentrated in certain local areas. The S/Ca and Al/Ca ratios of the inner and darker zone are similar to those of the inner products in the control mortar.

The S/Ca and Al/Ca ratios of the inner products formed during the heat cure and the outer products grouped together are further reduced for the heat-cured mortars that were stored for 1 year at room temperature (Fig. 12c). They approached those of the inner products formed during the room temperature cure whose S/Ca and Al/Ca ratios remained approximately constant throughout. It seems that less ettringite is distributed in the outer products than in the case of 90 days of water storage. Possibly some microcrystalline ettringite in the outer products dissolved and recrystallised in ettringite bands or ettringite-rich areas.

The results of X-ray microanalysis indicate that the sulfur and aluminum amounts in the inner products formed during the room temperature cure (the inner and darker zone) in the heat-cured mortars remained approximately constant and at a low level throughout the process of expansion. Therefore, there is little possibility for the cracks in the inner products formed at room temperature to have been produced by homogeneous expansion due to ettringite formation in this fraction of the inner products. The variations of sulfur and aluminum in the inner products during the whole period of expansion described by Lewis et al. [21] are in good agreement with the results for the inner products formed during the heat cure in the current work. Nevertheless, it seems to us that no compelling evidence can be found for ettringite formation in the inner products formed during the heat cure at any stage of the expansion process.

3. Conclusions

1. Quantitative XRD analysis for ettringite in the heat-cured mortars demonstrates that ettringite formation starts much earlier (about 7 days after heat curing) than the observed expansion (about 90 days after heat curing) and that it continues to increase during the period of DEF expansion. Hydrogarnet formation in the heat-cured mortar takes up part of the aluminum available and raises the potential of the system to form ettringite.
2. The microstructural observations of ettringite band formation in the expansive heat-cured mortars suggest that at least parts of these bands do not result from subsequent crystallisation of ettringite in cracks that were caused by a uniform expansion of the cement–paste matrix. It seems that ettringite band formation in heat-cured mortars can create, extend, and widen cracks, hence causing the overall expansion observed.
3. The results of EDS imply that a potential for DEF in the mortars heat cured at 100°C and subsequently

stored at room temperature is established as sulfate ions that were sorbed by C-S-H gel during the period of heat curing tend to be expelled. Driven by this potential, a portion of the late-forming ettringite develops as bands in the heat-cured mortar, instead of becoming intermixed with C-S-H gel as in the case of the mortar cured at room temperature, because of the dense microstructure of the heat-cured mortars that was already established during the period of heat cure. This kind of ettringite band development can cause expansion. Sulfur and aluminum analyses in the inner products formed at room temperature in the heat-cured mortars varied little with duration of water storage and had low levels close to those of the inner products in the mortar continuously cured at room temperature, throughout the period of expansion.

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