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A through-solution mechanism for delayed ettringite formation in pre-existing cracks in Portland cement mortar

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It has been reported that nucleation of ettringite crystals in cement pastes will preferentially occur in crack tip-zones rather than on plane solid surfaces in a cement-based matrix under conditions of supersaturation [1]. This explanation of the phenomena is compatible with results from many case studies where ettringite was usually found in cracks [2-4]. Recent research reported by Scrivener and Taylor [5] also confirmed this phenomenon. It was believed that high-temperature steam curing followed by high-temperature drying to form microcracks was an optimum condition for concrete deterioration due to secondary ettringite formation [6]. Two different arguments were discussed for interpretation of ettringite-related expansion mechanisms for Portland cement concrete: the crystal growth theory and the swelling theory. Cohen [7] postulated that expansion results from the growth of ettringite crystals formed on the surfaces of the Al-bearing particles. The growth of these crystals results in crystallization pressure and expansion occurs. Mehta [8] argued that expansion is attributed to the swelling of colloidal particles of ettringite. The formation of this swelling gel occurs by a through-solution mechanism. Expansion could occur either by intercalation or surface adsorption effects. The phenomenon of preferred nucleation of secondary ettringite in pre-existing cracks appears to support arguments for a through-solution deposition mechanism. Resulting expansion likely occurs through crystal growth in conforming with Cohen's postulation.

This letter provides further evidence in support of a previously reported study on nucleation characteristics of secondary ettringite in cracks of Portland cement paste [1, 6]. Secondary ettringite formation has been suggested as one of the critical mechanisms causing concrete deterioration. Test results presented here indicate that the reactants for secondary ettringite formation in cracks can come from sources away from the cracks. Diffusion of reactant ions through concrete solution appears to be a key element of the process responsible for initiation of secondary ettringite nucleation in cracks.

Materials used in this study include: type 50 Portland cement; Ottawa sand (ASTM C 109); reagent grade aluminium sulphate, $Al_2(SO_4)_3$; reagent grade calcium hydroxide, $Ca(OH)_2$; reagent

grade gypsum, $CaSO_4 \cdot 2H_2O$; and tricalcium aluminate, C_3A (supplied by Construction Technology Labs, Skokie, Illinois, USA). Two ettringite mixtures were prepared. One was prepared at 85 °C. The stoichiometric amount of reagent aluminium sulphate and calcium hydroxide required for ettringite formation was mixed with sufficient distilled water in a sealed plastic bottle. The mixture was reacted at 85 °C in a water bath for 30 days. The wet mixture was then vacuum dried for 48 h. A second ettringite mixture was prepared at 23 °C. The reactants and the preparation procedure were the same as the first.

The specimen design is shown in Fig. 1. The specimens were prepared in the following way: a cement mortar core assumed to contain no ettringite was enclosed in a cement mortar enclosure to which ettringite was added or formed. The mortar core was first cast in the shape of a 50 × 50 × 250 mm prism using type 50 Portland cement and Ottawa sand (ASTM C 109). The water-cement ratio was 0.5 and the cement-sand ratio was 1:2.75. A crack was carefully prepared by bending the prism specimen. The load was slowly increased until the crack extended about 75% of the specimen height so that the specimen was not broken. The specimen was then cut at a distance of 10 mm on each side of the crack. The thickness of the specimen was 20 mm. The specimen was sealed with epoxy at each end as shown. The mortar enclosure was made with the same material as the mortar core. Different additives however were incorporated into the mortar mixture to provide a source of ettringite. The additives are listed in Table I. The mortar enclosures

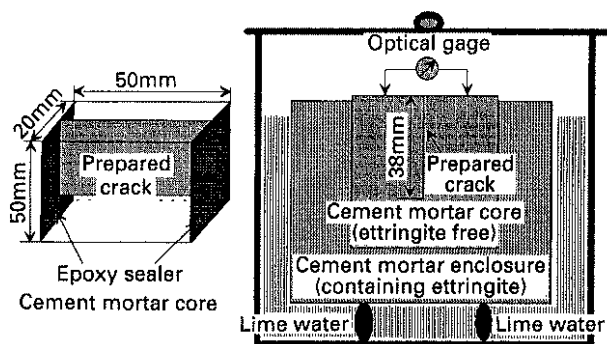


Figure 1 Schematic diagram of test specimens.

TABLE I Additives for mortar enclosure in test specimens

Specimen Designation	Additives (wt % of cement)
M-1	None
M-2	Ettringite formed at 85 °C (5%)
M-3	Ettringite formed at 23 °C (5%)
M-4	C ₃ A (10%) and gypsum (5%)
M-5	Al ₂ (SO ₄) ₃ (3%) and Ca(OH) ₂ (2%)

were mixed 28 days after the mortar cores. A mortar core hydrated 28 days with a prepared crack was embedded in each of the fresh mortar enclosures prepared with a different formulation. The crack mouth was placed above the surface of fresh mortar so that the crack would not be contaminated by ettringite. The composite specimen (core + enclosure) was cast in a 50 × 50 × 50 mm mould. The specimens were demoulded after 24 h hydration. The specimens were immersed in lime water up to 80% of the height of the specimens. The open mouth of the crack was kept out of the lime water. The ions could only diffuse into the crack from the two sides perpendicular to the crack. An optical strain gauge was mounted across the crack mouth. The dimensional change due to the opening of crack mouth was recorded. The crack mouth opening was attributed to ettringite formation in the crack. In addition to the preparation of cubic specimens with a cracked ettringite-free core, specimens made from only mortar enclosure material were also cast as prisms, 25 × 25 × 125 mm. The length change was recorded from 1 day to 180 days.

The expansion results of the enclosure mortars is shown in Fig. 2. It is apparent that little expansion occurred in the specimens containing prepared ettringite (M-2 and M-3) and the control specimen. Expansion resulted at early ages only (before 14 days) for the enclosure mortar specimens containing reactants required for *in situ* formation of ettringite.

Expansion at the crack open mouth is shown in Fig. 3. No significant expansion was found for control sample (M-1) and the specimen containing ettringite prepared at 23 °C (M-3). The specimen containing ettringite prepared at 85 °C (M-2) started to expand appreciably after 60 days. Its expansion was about 0.13% at 180 days. The specimens M-4 and M-5 expanded much earlier than specimen M-2. The expansion started to accelerate at about 40 days. Their expansion values at 180 days were 0.09% and 0.11% for M-4 and M-5, respectively.

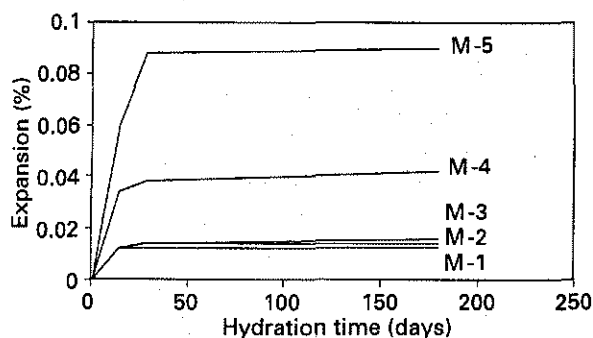


Figure 2 Expansion of enclosure mortars.

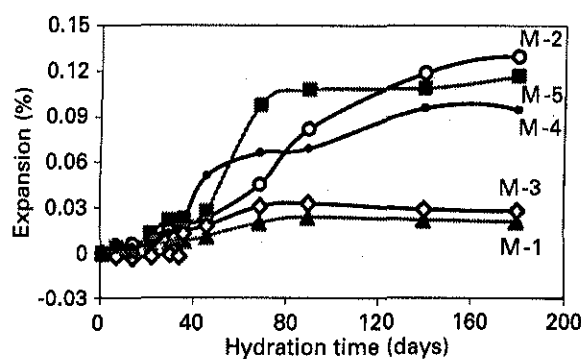


Figure 3 Expansion at the open mouth of a pre-existing crack in Portland cement mortar surrounded by mortar containing ettringite or sources of ettringite formation (▲ M-1; ○ M-2; ◇ M-3; ● M-4; ■ M-5).

Change in the crack mouth opening is attributed to ettringite formation in the cracks (Fig. 4). This appears to constitute further evidence that ettringite can preferentially crystallize in the crack, cause extension of the crack and ultimately lead to damage of cement paste. The crystallization rate is dependent on the degree of supersaturation of the reactants required to form ettringite nuclei. These reactants can only come from the enclosure mortar and by diffusion through the ettringite-free cement mortar wall. The concentrations of reactants (Al³⁺, Ca²⁺ and SO₄²⁻) in the specimens M-4 and M-5 during the process of ettringite formation might be higher than that in the equilibrium system containing pre-formed ettringite. A high reactant concentration results in a high diffusion rate of reactant ions. This accelerates crystallization and subsequent expansion. It is apparent that ettringite formation in the crack occurs by a through solution mechanism. Stages in the deterioration of concrete due to secondary ettringite formation may be described as follows:

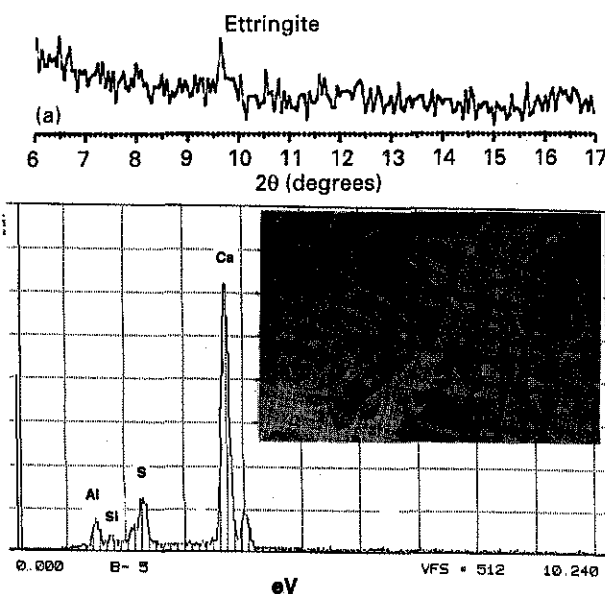


Figure 4 Ettringite formation on the surface of a pre-existing crack. (a) X-ray diffraction spectrum of the crack surface of type 50 Portland cement mortar core. (b) Photomicrograph from the SEM and EDXA investigation of the crack surface of type 50 Portland cement mortar core.

(1) *Presence of internal sulphate sources* High adsorption of sulphate by C-S-H gel cured at high temperature and slow desorption of sulphate at later ages are processes that can serve as internal sulphate sources for secondary ettringite formation [9, 10].

(2) *Diffusion of reactants into pre-existing cracks through concrete solutions* Water curing accelerates secondary ettringite formation by providing a medium for diffusion of reactants, i.e. satisfying the requirements for occurrence of a through solution process [11].

(3) *Preferred crystallization of ettringite in cracks* This results in the extension of cracks and the damage of concrete.

Further evidence that ettringite preferentially crystallizes in pre-existing cracks is provided by this study. Diffusion of reactants from sources elsewhere in concrete into pre-existing cracks appears to follow a through-solution mechanism.

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