

Microstructure of Highly Expansive Cement Pastes



by Yan Fu, Shamim A. Sheikh, and R. Douglas Hooton

An investigation at the microstructural level was carried out to interpret the mechanisms of hydration processes and the properties of highly expansive cement pastes before and after set. Results from this investigation, which included analyses from x-ray diffraction (XRD), mercury intrusion porosimetry (MIP), and scanning electron microscopy (SEM), are presented. Some crucial behavior occurring in the hydration of expansive cement paste, such as quick setting and adverse effects of admixtures on expansion properties, are discussed. The mineral composition of such material is also studied with respect to its durability.

Formation of calcium sulfoaluminate immediately after the solid materials are mixed with water was observed to be mainly responsible for the quick setting behavior of expansive cements containing high-alumina cement (HAC). Use of hydrated HAC instead of HAC as the Al-bearing material delayed the formation of sulfoaluminate (SA), resulting in a more workable cement. The superplasticizer accelerated the formation of SA in fresh expansive cement paste.

Keywords: cement pastes; expansive cements; ettringite; high-alumina cements; microstructure; x-ray diffraction.

A special concrete has been developed to produce free expansion exceeding 4 percent for use in drilled shaft foundations¹ to create a stronger bond between the shaft concrete and surrounding geomaterial and enable the foundation to carry a substantially higher load² than would be possible by using normal concrete. Further research has been completed to modify this concrete to extend the setting time and evaluate the effect of these modifications on engineering properties.* Simultaneously, a study of the mechanisms of expansion and microstructure of the paste component of this expansive concrete was undertaken and is reported here. This was considered necessary because the magnitudes of expansion of the cements discussed here are much higher than those in more conventional shrinkage-compensating or mildly expansive cements.³ In previous research on mechanical properties,³ some factors, such as water-cement ratio (w/c), admixtures, and type of Al-bearing materials, have been recognized as important parameters dramatically affecting the properties of expansive concrete. For its use in the field at this stage, it was difficult to predict concrete behavior if the field operations were not strictly carried out according to the pre-

scribed procedure. Therefore, an investigation at the microstructural level helps to clarify the relationship between different variables and actual behavior of the material with respect to setting, expansion, and strength characteristics.

It is difficult to interpret entire mechanisms of the hydration reactions in this type of highly expansive cement paste, because this involves a number of chemical reactions taking place simultaneously. However, it can be stated that almost all the factors affecting the properties of such materials relate to the formation and growth of ettringite, which is the major focus of attention in this research.

The microstructural study of expansive cement paste involves: 1) microstructural characteristics of the cement during setting and expansion and 2) mechanisms of formation of hydration products that govern the properties of fresh and hardened cement paste. Testing included x-ray diffraction (XRD), mercury intrusion porosimetry (MIP), and scanning electron microscopy (SEM).

TEST PROGRAM

Three instruments, an x-ray diffractometer (using copper $K\alpha$ radiation), a scanning electron microscope, and a mercury intrusion porosimeter (with contact angle assumed to be 140 deg), were used in this test program to measure: a) the change of mineral composition during hydration, b) the morphology of expansion cracks and hydration products, and c) the pore size distribution in expansive cement pastes.

The raw materials used in this test program were the same as those used for the tests described in the earlier study.* These commercially available materials were: ordinary portland cement (OPC, ASTM Type 1), high-alumina cement (HAC), quick-set plaster ($\text{CaSO}_4 \cdot 0.5 \text{H}_2\text{O}$), and hydrated lime ($\text{Ca}(\text{OH})_2$). The admixtures included retarders (such as sodium citrate), a naphthalene sulfonate condensate powdered

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superplasticizer, and a Class F fly ash. To help control setting and expansion rate in some mixtures, the HAC was prehydrated for 1 day, then dried and crushed until the particle size varied from 75 to 150 μm in diameter. The mixtures designed for the current test series are as follows and are detailed in Table 1.

1. Reference HAC (high-alumina cement)-type expansive cement paste without superplasticizer or retarder (M-1)
2. HAC-type expansive cement paste with retarder and superplasticizer mixed by one-stage mixing process (M-2)
3. Identical to No. 2, but mixed by a two-stage mixing process (M-3)
4. H-HAC (hydrated high-alumina cement)-type expansive cement paste without superplasticizer or retarder (M-4)
5. H-HAC-type expansive cement paste with superplasticizer (M-5)

In the one-stage mixing process, all materials were mixed together. In the two-stage mixing process, normal portland cement paste is prepared first, then, after 5 min, the expansive components are mixed in. The water-to-solid content was kept constant at 0.43 for this study.

Preparation of samples and tests

Two types of samples were prepared in these tests:

Fresh paste samples—To measure the hydration process of expansive cement paste in the fresh state, two grams of solid materials were mixed continuously and uniformly with water or water-admixture solution in a glass beaker at 23 C. The hydration periods were fixed at 1, 3, 5, 10, 20, 30, and 60 min. At the designated time, the hydration of the fresh paste

was terminated by adding 30 ml of propanol, and then the samples were filtered in a funnel with qualitative filter paper (Grade 601-25). After washing the sample with propanol three times on the filter paper, the remnant on the paper was dried in a vacuum desiccator with a negative pressure of 100 kPa for 48 hr. Then the sample was ground in an agate mortar with 10 percent CaF_2 as an internal standard.

Hardened specimens—To explore the microstructure of expansive cement paste, the specimens must be prepared under conditions simulating those in real drilled shafts. Cylindrical specimens 26 mm in diameter and 50 mm high were cast in small steel tube molds with 2-mm thick walls. Two 8-mm thick steel plates were placed at each end of the steel mold and tightly screwed together using three 6-mm-diameter threaded rods. At each end of the mold, there were two holes (each 3 mm in diameter) for water supply during hydration. In the first 24 hr, the specimens were cured in air at 100 percent relative humidity (RH) and 23 C, and then stored in 23 C tap water. During the entire curing process, the specimens were subjected to three-dimensional restraint. At designated ages, the threaded rods were removed and the steel molds were cut longitudinally to obtain the paste cylinder specimens. This procedure was followed to avoid damage to the paste during demolding. Hardened paste from the central section of the specimen was then taken and crushed into 3- to 10-mm-diameter particles. The samples were immersed in propanol for 24 hr to terminate hydration and then dried in a vacuum desiccator at a negative pressure of 100 kPa for 48 hr. The particles were then ground and sieved into three grades: particles with diameter of about 10 mm were chosen for scanning electron microscopy; particles with diameter of about 2 to 3 mm were favored for porosimeter tests; and the rest of the material was ground together with 10 percent CaF_2 as an internal standard for x-ray diffraction tests.

TEST RESULTS

X-ray diffraction analysis

X-ray diffraction patterns of five expansive cement pastes during hydration in the first 60 min and at later ages are presented in Fig. 1 and 2, respectively. Calculated from these figures, the relative intensities of three designated minerals, gypsum (G), hemihydrate (B), and calcium sulfoaluminate (SA), are presented in Fig. 3 and 4. Calcium sulfoaluminate (SA) is used here to designate the combination of monosulfoaluminate ($\text{C}_3\text{A} \cdot \text{CS} \cdot 12\text{H}$) and ettringite ($\text{C}_3\text{A} \cdot 3\text{CS} \cdot 32\text{H}$). With hydration, the intensities of SA obviously increased. In

Table 1 — Proportions of expansive cement formulations (units: mass)

Sample	Ordinary portland cement	Expansive components				Admixture		Water	Type of mix process
		Al-bearing material		S̄-bearing material	Hydrated lime				
		HAC	H-HAC	Quick-set plaster		Lomar-D	Sodium citrate		
M-1	0.6	0.25	—	0.12	0.03	—	—	0.43	One-stage
M-2	0.6	0.25	—	0.12	0.03	0.015	0.000375	0.43	One-stage
M-3	0.6	0.25	—	0.12	0.03	0.015	0.000375	0.43	Two-stage
M-4	0.6	—	0.25	0.12	0.03	—	—	0.43	One-stage
M-5	0.6	—	0.25	0.12	0.03	0.0075	—	0.43	One-stage

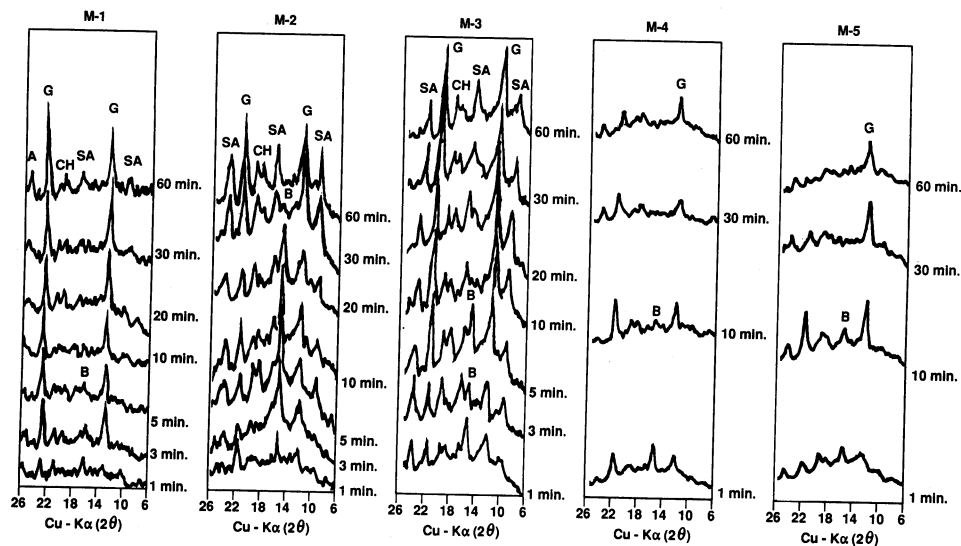


Fig. 1—X-ray diffraction patterns of expansive cement pastes during first 60 min of hydration

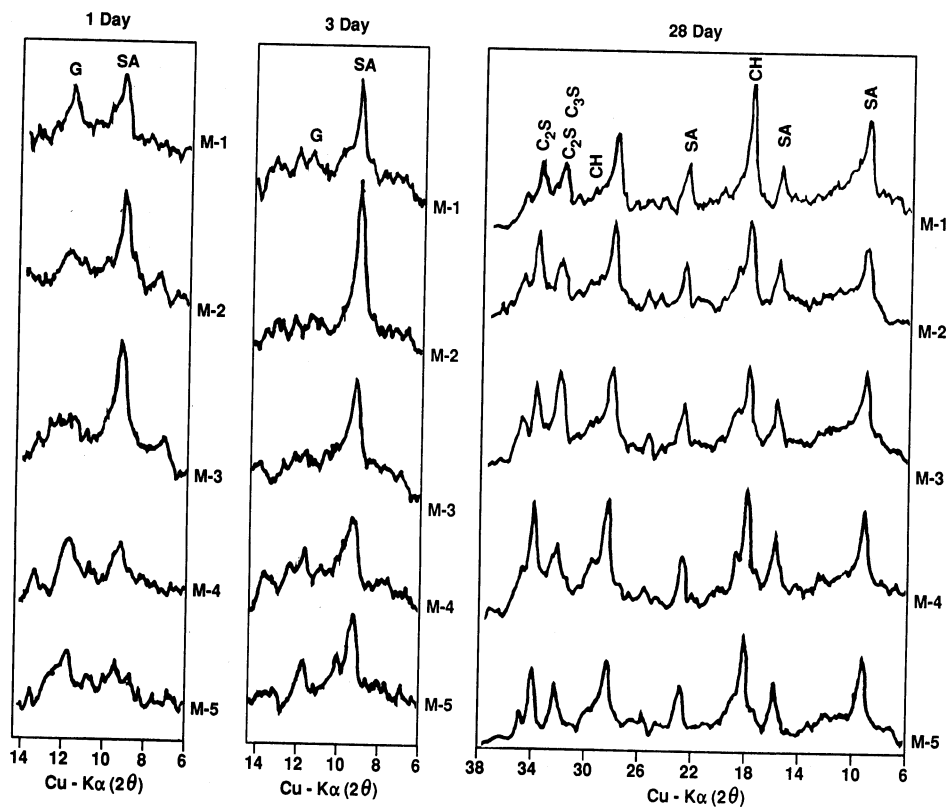


Fig. 2—X-ray diffraction patterns of expansive cement pastes at different ages

samples with admixtures, the increase in SA intensities at an early age was larger than in the sample without admixtures. In HAC-type expansive cement (M-1, M-2, and M-3), the hemihydrate peak diminished expeditiously and disappeared before 60 min with a corresponding increase of the gypsum peak. In the H-HAC-type expansive cement (M-4 and M-5), the hydration rate seemed very slow. Hemihydrate existed in the pastes for 24 hr and the intensity of SA was quite small in the first 60 min even with admixtures. With the increase of SA formation, gypsum was greatly consumed, which resulted in the decrease of gypsum's relative intensity at later ages

(Fig. 4). Since the same content of plaster (hemihydrate) was used in all the mixtures, the relatively lower gypsum intensity corresponded mostly with a high rate of SA formation. The peak shift from 9.90 to 9.05 deg 2θ as hydration proceeded showed the composition change of calcium sulfoaluminate from monosulfoaluminate to ettringite, which will be discussed later. The patterns of cement pastes with admixtures exhibited a distinct background hump due to amorphous phase in the range of 8 to 25 deg 2θ (Fig. 1), indicating acceleration of the hydration rate.

In the x-ray diffraction analyses of expansive cement pastes

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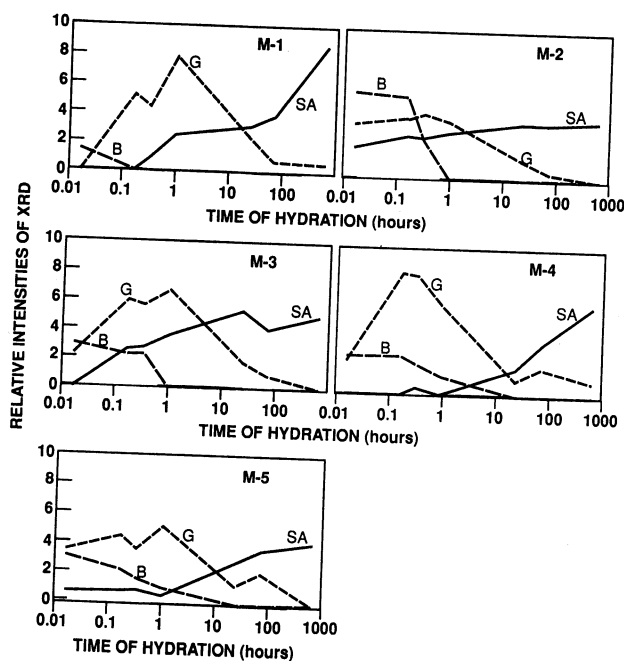


Fig. 3—Relative intensities of sulfoaluminate (SA), gypsum (G), and hemihydrate (B) of expansive cement pastes

after 1 day, it was obvious that the sulfoaluminate peak of the HAC-type expansive cement with admixtures was higher than that of the reference expansive cement, but no obvious difference could be found between two samples with different mixing processes (M-2 and M-3). In the H-HAC-type expansive cement pastes, the SA peaks were much lower than that in the HAC-type expansive cement, indicating a reduced rate of SA formation.

At 3 days, the x-ray diffraction pattern of expansive cement pastes was similar to those at 1 day. But the relative intensities of designated minerals (SA, G, or B) in different expansive cement pastes became somewhat similar.

At 28 days, the five expansive cement pastes were not discernably different even in the intensity of each peak. The major hydration products were ettringite and calcium hydroxide, while some unhydrated clinker phases such as C_3S and C_2S were also noted. However, no characteristic peaks representing the hydration products of HAC, such as C_3AH_6 , could be detected.

Morphology of ettringite and microstructure of expansive cement paste under SEM

The expansion cracks existing in various expansive cement pastes with different admixtures and curing ages are shown in Fig. 5. Under the particular restraint conditions in the steel mold, the widths of cracks varied from 10 to 30 μm and they increased with an increase in age at early ages, but then decreased dramatically at 28 days [Fig. 5(e)]. The surface texture of the 28-day specimens was dense, and most remaining microcracks were closed. At early ages, they were quite porous and the cracks extended through the sample.

At early ages, average crack widths of the HAC-type expansive cement pastes with admixtures [Fig. 5(b)] were larger than those in the pastes without admixtures [Fig. 5(a)], while in the H-HAC-type expansive cement pastes [Fig. 5(d) and ACI Materials Journal / January-February 1994

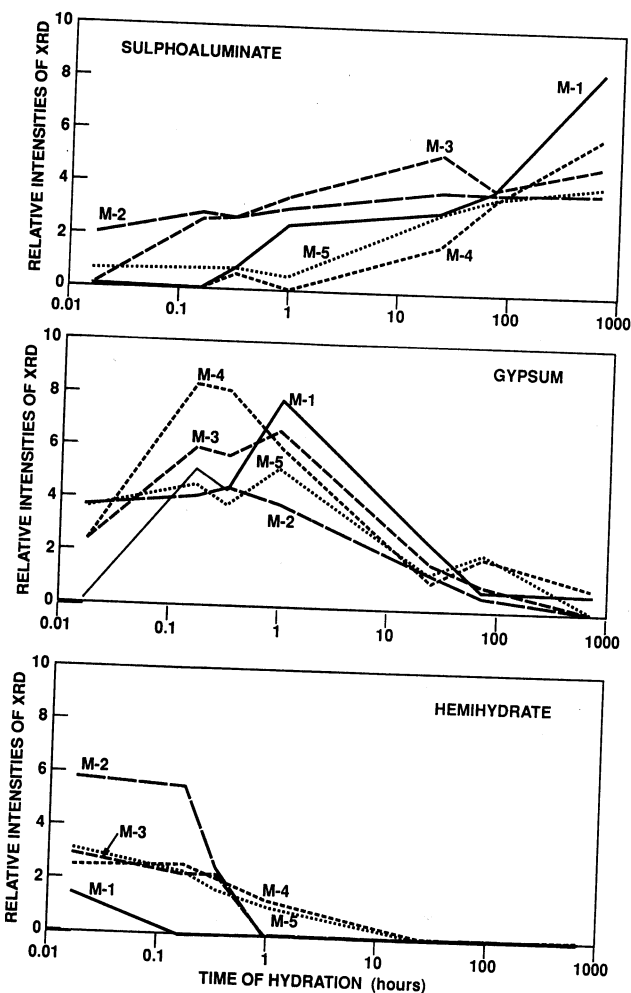


Fig. 4—Comparison of relative intensities of sulfoaluminate (SA), gypsum (G), and hemihydrate (B) in various expansive cement pastes

(e)], the crack widths were only about one-fifth of those in the HAC-type expansive cement pastes. At 28 days [Fig. 5(e)], the surface morphology was totally different from those at early ages. Only traces of sulfoaluminate crystals could be found in some isolated areas.

The morphologies of SA in pastes at different ages are demonstrated in Fig. 6. It is interesting to note that in the HAC-type expansive cement paste without admixtures [Fig. 6(a)], large-size SA crystals (about 15 to 60 μm long and 6 μm in diameter) were commonly found. But in the HAC-type expansive cement paste with admixtures [Fig. 6(b)], only small needle-shaped SA crystals existed in clusters. The smaller crystals were about 3 to 5 μm long and 1 μm in diameter. Although in the H-HAC-type expansive cement paste, some SA crystals located in pores or cracks were large, about 10 to 20 μm long and 2 μm in diameter [Fig. 6(c)], most other crystals were still smaller than those observed in the HAC-type cement pastes with admixtures [Fig. 6(b)].

At early ages, most of the SA crystals appeared as conglomerations irregularly interlocked with each other, and those in the pores or cracks grew from solid side surfaces into the open space. At 28 days, ettringite could not easily be detected or identified because of the extreme dense structure of the hardened paste [Fig. 6(d)]. Occasionally, some small clus-

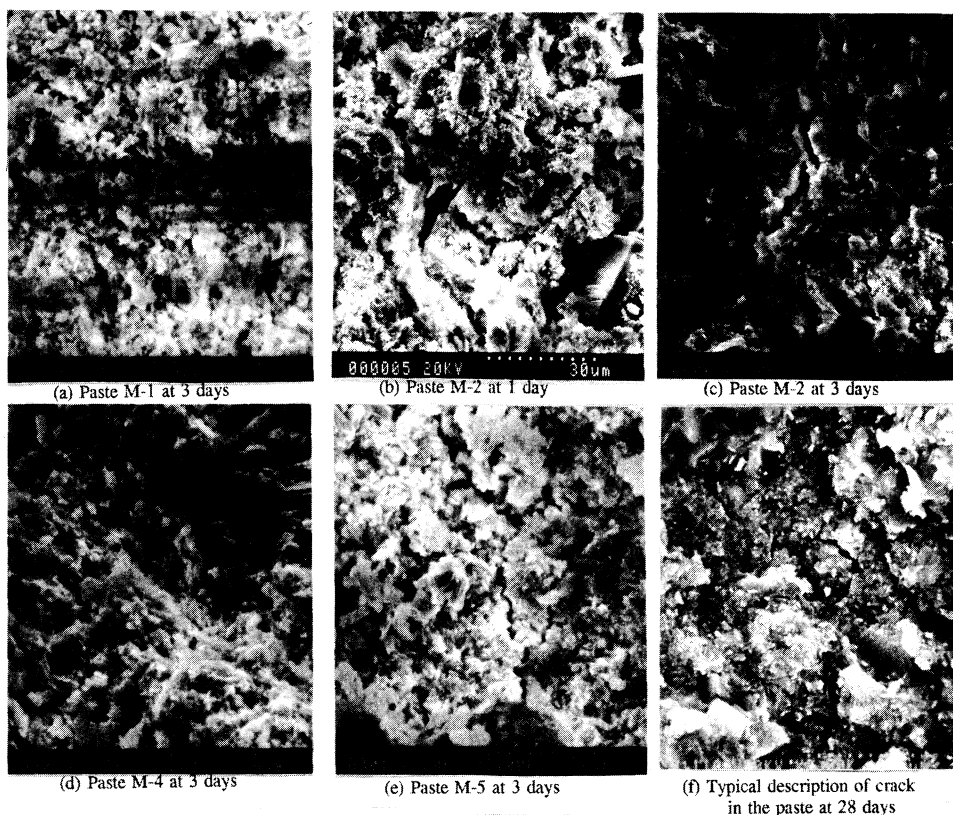


Fig. 5—Description of cracks in expansive cement pastes

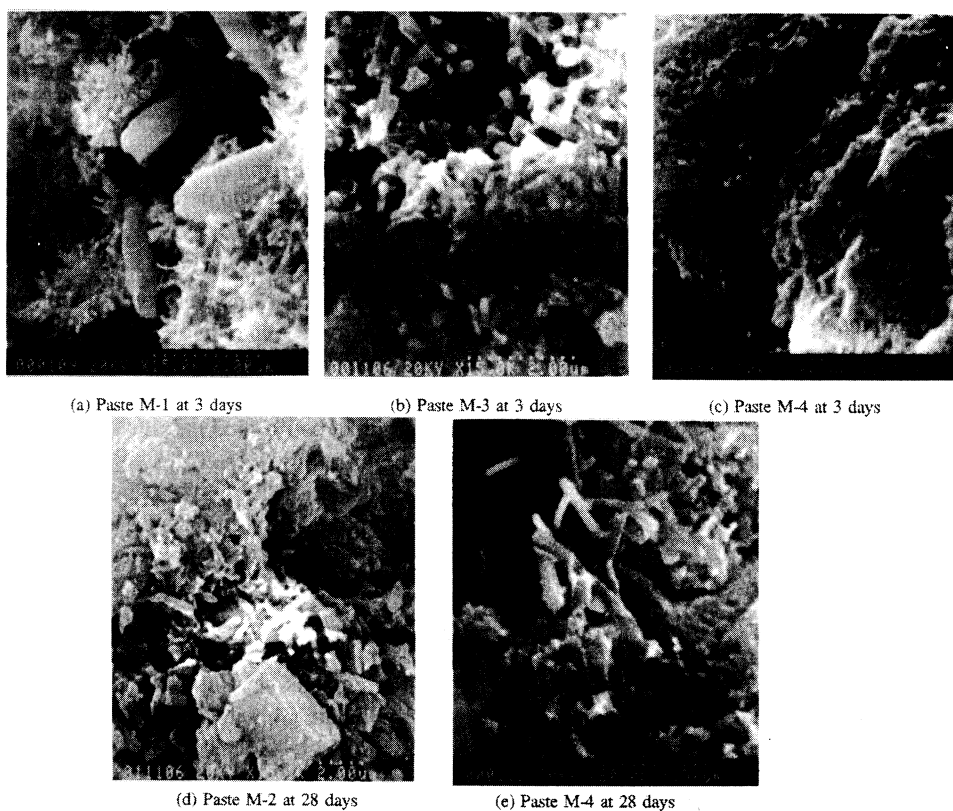


Fig. 6—Morphology of sulfoaluminate crystals

ters of ettringite could be found in the pores or some weak areas [Fig. 6(e)].

Pore structures of hardened pastes

Cumulative pore size distribution curves of the expansive

cement pastes are shown in Fig. 7. With hydration, cumulative pore volumes at all sizes decreased in all the pastes. At 28 days, the volume of pores larger than 400 Å tended to be zero. This indicated that hydration products had filled in the pores. The total pore volume of H-HAC-type cement pastes

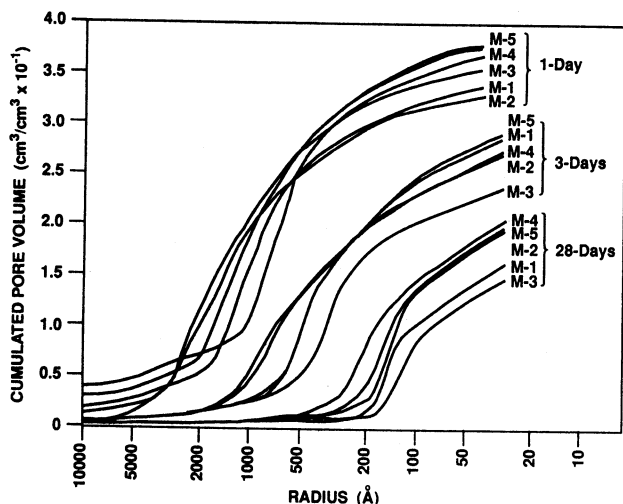


Fig. 7—Cumulative pore size distribution curves of expansive cement pastes

was slightly higher than that of HAC-type expansive cement pastes.

Fig. 8 shows the differential pore size distribution curves at different ages for expansive cement pastes. The most probable pore sizes (MPPS) of these samples at 1 day [Fig. 8(a)] were in the range of 600 to 3000 Å. Two samples of expansive cements without admixtures (M-1 and M-4) had the same MPPS of 3000 Å. As a result of using admixtures, the MPPS shifted to smaller sizes. At 3 days of hydration [Fig. 8(b)], the MPPS range for these samples was between 300 and 900 Å. The effect of admixtures on the character of distribution curves was the same as that at 1 day.

Fig. 8(c) demonstrates differential pore size distributions of 28-day pastes. The MPPS were very low, from 80 to 160 Å. The MPPS of the H-HAC-type expansive cement paste was larger than that of HAC-type paste. This large reduction in pore volume is not common even in normal cement pastes incorporating silica fume, indicating that, when restrained, expansive cements at later ages would have an extremely dense structure and high strength.

DISCUSSION

Mechanism of setting in improved expansive cement paste

From the x-ray diffraction test results, the major changes in the mineral composition during the setting period appear to involve two types of hydration reactions:

Sulfoaluminate formation—SA formation starts immediately after the solid materials are mixed with water, and then it grows steadily. By noticing the variation of SA characteristic peaks in the range from 9.90 to 9.05 deg 2θ, representing the monosulfoaluminate (MS) and ettringite, respectively, it can be stated that with the passage of the hydration, the SA peak shifts to a lower d-spacing, indicating a composition change from the lower sulfate state (MS) to the higher one (ettringite). A small hump combining two peaks between 9.90 and 9.05 deg 2θ exists at early ages, but at 28 days, they join together into one higher peak at 9.05 deg 2θ, where only ettringite exists as the final state of SA in the paste. Actually, ettringite is not formed in one step, but is converted from

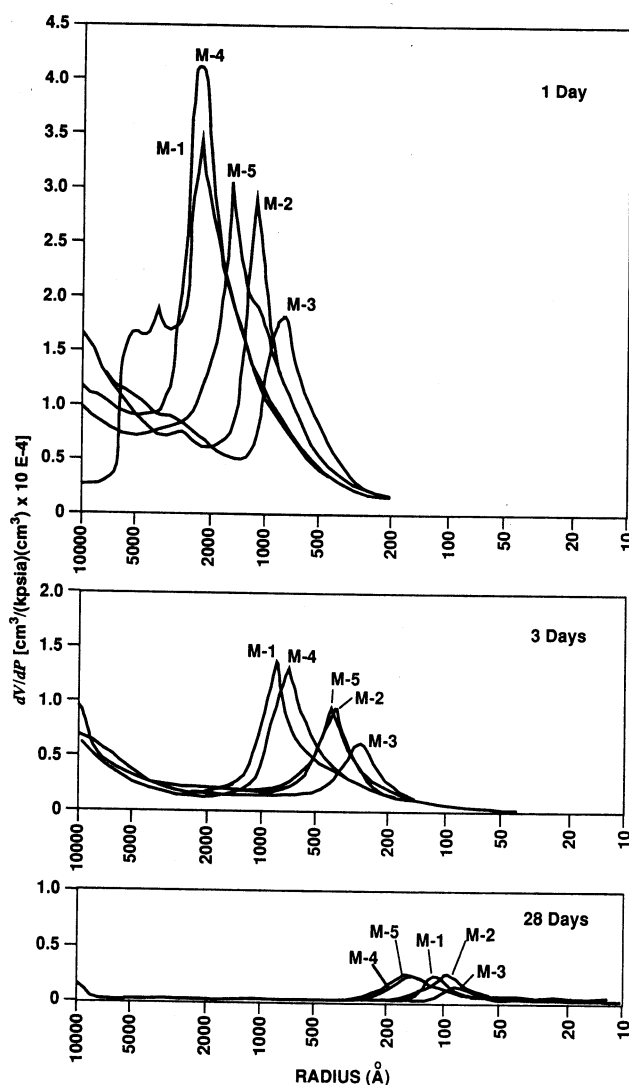


Fig. 8—Differential pore size distribution curves of pastes at different ages

monosulfate. Initially, MS is most easily formed in the structure, and then, with sufficient gypsum, CH, and water, the monosulfate will combine more sulfate and water to form ettringite. During the period of hydration, various states of SA can be present in the paste at the same time. This phenomenon has been reported by Cohen.⁴

Hemihydrate converts to gypsum—In this research, because hemihydrate was used as the sulfate-bearing material, it can hydrate immediately to form gypsum with two molecules of water. From the x-ray diffraction pattern, the increase of the gypsum peak is the most distinct change during hydration in the first 60 min.

Two identical effects of the preceding two reactions on the setting behavior of expansive cement pastes can be observed: 1) consumption of a large amount of free water and (2) formation of needle-like crystals. In the H-HAC-type expansive cement, although the SA formation at early ages is limited, the conversion reaction from hemihydrate (B) to gypsum (G) proceeds at almost the same rate as that with the HAC-type expansive cement. So it can be concluded that the formation of gypsum is responsible for the false set. SA formation dominates the setting behavior in the expansive pastes.

Adverse effect of admixtures on expansion

For HAC-type expansive cement, admixtures do not suppress the formation of SA but act as an accelerator, so the idea that the retarder and water reducer will restrict the rate of ettringite formation is debatable. The question, however, is why this accelerator can delay the setting time even marginally, as observed in the physical tests.³ From SEM investigation, it has been found that, because of the action of admixtures, the size of SA crystals becomes smaller and the hydrates are distributed more uniformly in the paste. In fact, the reaction solids are better dispersed and the effective reaction area is increased. The delay of setting time is controlled by the high content of superplasticizer, which can efficiently separate the new products and keep the particles away from each other, forming a stable electric double-layer system on the particle surfaces and maintaining a stable flow in the fresh paste. Although this separation improves the setting behavior of the paste, the superplasticizer accelerates the SA formation such that a large amount of expansive material is wasted before the paste hardens. Therefore, a large number of SA crystal nuclei are uniformly distributed in the paste and act only as an efficient filler to make the paste dense.

In the H-HAC-type expansive cement, the SA peak at early ages is very small, so not much expansive material is wasted prior to setting.

Mineral composition of expansive cement paste

From the x-ray diffraction analysis of 28-day pastes, background humps indicate that poorly crystallized C-S-H gel also exists in the expansive cement paste matrix, similar to that in portland cement pastes, and is the fundamental contributor to strength. An interesting phenomenon shown in this analysis is that no clear peak representing the hydration products of HAC can be detected, and at 28 days, the gypsum has almost been consumed, showing a simple mineral composition in the expansive cement paste: ettringite, calcium hydroxide, C-S-H, and some unhydrated portland cement. The HAC is the source of ettringite and is a convenient material for design of an expansive concrete. The hydration of HAC does not follow the traditional production of calcium aluminate, but forms ettringite if the conditions are suitable.

Mechanism of ettringite formation in drilled shafts

From the x-ray diffraction results, three conditions necessary for ettringite formation are suggested:

Sufficient supply of sulfate—As discussed previously, if the Al-bearing material has been completely hydrated, but the gypsum is not totally consumed, the conversion of monosulfaluminate to ettringite will still be completed. Gypsum can be detected in the specimens at 28 days. This explains why the expansive cements may have an extended period of expansion after 28 days.

Sufficient water supply—Under lab conditions, because of the small size of specimens and sufficient water curing, there is no differential water content from outside surfaces to the center of the specimens. But such differences may occur in field applications such as in drilled shafts, causing nonuniform hydration or ettringite formation. Sufficient hydration will cause a decrease in the porosity of the cement paste according to the pore size distribution results. Therefore, in the

outer part of a shaft, the concrete would experience better curing than in the center, and this denser concrete layer would decrease the water permeability to lower the subsequent hydration rate of the interior. In the outer part of the shaft, where sufficient water can be easily obtained, ettringite will form quickly and the corresponding expansion would occur earlier, but in the center of the shafts, expansion would develop at a slower rate and take longer until the ultimate stage, when all MS is converted into ettringite. This delay in expansion has been observed from comparing the test results obtained in a recent study³ with those reported by Sheikh and O'Neill.⁵

Temperature—Research reported by Heinz and Ludwig^{6,7} indicates that the temperature can influence formation of SA and its composition. When the temperature exceeds 70°C, the MS is more stable, but at room temperature ettringite becomes stable. So when MS is formed at a higher temperature, it may convert to ettringite when exposed to lower temperatures later on. Measurements on the heat of hydration of expansive cement pastes indicate that at an age of about 24 hr, temperature will increase to a maximum value of about 50°C for a 0.005 m³ insulated specimen. With a large volume of concrete, it is possible that the temperature in the center of drilled shafts would exceed 70°C. So at early ages of an expansive concrete drilled shaft, the SA most likely would be in the form of MS. At later ages, after the temperature drops, the MS would start to convert to ettringite. This can be another cause of prolonged expansion periods in shafts. Thus, the hydration heat affects expansion in two different ways: 1) accelerating the formation of SA; 2) leading to more MS formation rather than ettringite at early ages. After sufficient hydration, the porosity will be lower because of the hydration of portland cement. When the space, which is available to be filled by ettringite converted from MS, decreases, the products involved in this ettringite formation can work more effectively in creating expansion pressure.

Pore structure of expansive cement paste and its effect on expansion and durability

From the pore size distribution results, when using superplasticizer, the cumulative pore volume and MPPS clearly decrease at both 1 and 3 days. Under identical conditions, with the same content of portland cement and w/c, the degree of hydration of portland cement should be the same in all samples. The only contribution to higher densification in some samples is, therefore, because of more ettringite formed in the pores. Although this phenomenon may help enhance the early strengths of the paste, it would result in wasting a considerable amount of expansive material.

SEM pictures of 28-day pastes reveal that some microcracks still remain in the specimen with a width of several microns. But in porosimeter analysis for the same specimens, pores of such size were all but eliminated by 28 days (Fig. 8), indicating that most of these microcracks have been closed by new hydration products. The cracks, if they still remain in the paste at late ages, are only closed pores which should not significantly influence the mechanical properties and durability of the expansive concrete.

CONCLUSIONS

From the x-ray diffraction analysis, two major changes in mineral composition are apparently responsible for the set-

ting behavior of expansive concrete: 1) calcium sulfoaluminate (SA) formation starts immediately after the solid materials are mixed with water and then its volume increases steadily; 2) the hemihydrate added in the expansive component converts to gypsum. These reactions have identical effects in shortening the setting time by consuming a large amount of free water and forming needle-like crystals. Compared with the HAC-type expansive cement paste, the rate of formation of SA in H-HAC-type expansive cement paste is very slow at early ages, so the latter does not show a quick setting behavior. The superplasticizer does not suppress the formation of SA in fresh expansive cement paste, but rather acts as an accelerator. By separating hydration products as they form, it avoids their interlocking and delays the setting time somewhat. This accelerating action of the superplasticizer on the SA formation is the main reason for the adverse effect of such admixtures on expansion properties.

At 28 days, the mineral composition of expansive cement paste in all the mixtures includes: ettringite, calcium hydroxide, C-S-H gel, and some unhydrated portland cement. No clear XRD peak representing the hydration products of HAC can be detected at this stage, indicating that the hydration of HAC does not follow the traditional formation of calcium aluminate hydrates, but produces monosulfoaluminate or ettringite directly. The structure of the restrained expansive cement paste at this stage is dense, and cracks due to expansion, if they still remain, become closed pores which should

not significantly influence the mechanical properties and durability of the expansive concrete.

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