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Studies on delayed ettringite formation in early-age, heat-cured mortars
I. Expansion measurements, changes in dynamic modulus of elasticity, and weight gains

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Abstract

Mortars were prepared from laboratory cements blended from a set of six representative ground clinkers and Terra Alba gypsum. The addition of gypsum was such that cements containing 1% SO_3 less than the optimum SO_3 content, the optimum SO_3 content, and 1% greater than the optimum SO_3 content were produced. Mortar bars and mortar cubes containing each of these cements were exposed to continuous room temperature (23 °C) curing, or to early-age curing cycles involving maximum temperatures of 55 and 85 °C, followed by long-term exposure at 100% RH over water, but not immersed in water. Measurements of expansion, dynamic elastic modulus, and weight gain were recorded at intervals of up to 900 days. Severe cracking and prominent delayed ettringite formation (DEF)-induced expansions were observed in 85 °C cured mortar bars derived from four of the six "oversulfated" cements. Much smaller expansions were observed in mortar bars from two cements with optimum SO_3 content cements also cured at 85 °C. No expansion or other visible indication of distress was observed for any of the 55 °C or continuously room-temperature-cured mortars. The dynamic elastic modulus increased progressively on prolonged exposure for the unaffected mortar bars, but it decreased precipitously after the onset of expansion in affected mortar bars. Significant weight increases also accompanied the processes of expansion. Mortars that showed severe cracking and deterioration when exposed as mortar bars suffered almost no visible damage when exposed as cubes.

Keywords: Delayed ettringite formation (DEF); Optimum sulfate content; Expansion; Mechanical properties; Elastic modulus

1. Introduction

Despite a large number of laboratory studies on delayed ettringite formation (DEF), in laboratory mortar bars and cement paste prisms (e.g., Refs. [1–10]), there is still considerable uncertainty on a number of points. These include criteria for distinguishing cements which may give rise to DEF problems from those that appear to be immune, the DEF mechanism itself, and the relevance of laboratory mortar bar studies of DEF to the actual deterioration processes taking place in DEF-affected field concrete. The authors hope that the results and interpretations presented in this and subsequent papers in this series will help clarify these important questions.

2. Cement selection and sulfate level

In the present research, an attempt was made to relate the DEF processes to cement chemistry. In particular, the relevance of sulfate content to whether or not a particular cement would be susceptible to DEF was of immediate concern because of its obvious importance to the cement industry. The sulfate content is viewed here not in terms of its absolute numerical value, but with reference to the "sulfate demand" of the particular clinker involved, as suggested by its optimum SO_3 content as established in accordance with ASTM C 563.

Accordingly, the suite of cements to be studied here was assembled in a manner somewhat different than those used in most laboratory DEF studies.

A group of six ground clinkers, of widely varying composition but with variations within the normal range of compositions for American portland cements, was selected and obtained through the courtesy of Construction

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Technology Laboratories (CTL). The clinker sulfate contents varied from practically nil (0.07%) to almost 2.5%. The alkali contents also varied widely (from 0.22% to as much as 1.15% $Na_2O_{equiv.}$). The Bogue calculated C_3A contents varied from as little as 0.4% to almost 15%. The analyses of each of these clinkers, together with the calculated Bogue contents of C_3S, C_2S, C_3A, and C_4AF, are provided in Table 1.

The specific clinkers used may variously be described as follows. Clinker No. 1 is a high-alkali, low-sulfate clinker; Clinker No. 2 is a very-low-alkali and very-low-sulfate clinker; Clinker No. 3 is a high-alkali, high-sulfate clinker with a high C_3A content ; Clinker No. 4 is a low-alkali clinker with a very low C_3A content, suitable for a Type V (sulfate-resisting) cement; Clinker No. 5 is a very-high-alkali clinker with a very high C_3A content and a moderate sulfate content; and Clinker No. 6 is high-alkali, high-sulfate clinker. In these descriptions, where C_3A content is not specifically mentioned, it is considered to be moderate.

Each of these clinkers had been ground by CTL to a uniform Blaine fineness level of 350 m^2/kg.

The optimum cement sulfate content was determined for each of these clinkers, as specified in ASTM C 563. In this procedure, the "optimum SO_3" content is the total % SO_3 (including the amount present in the clinker and the amount added as gypsum) that produces the highest 24-h compressive strength at 23 °C. Most commercial cements in the US and elsewhere have SO_3 contents close to the optimum value for the specific clinkers employed. In this study, the gypsum used in formulating the final cements was a high-purity Terra Alba gypsum of approximate Blaine fineness of 900 m^2/kg.

The optimum SO_3 contents as determined in this laboratory for the clinkers used are also provided in Table 1. The values ranged between about 2.8% to about 4.0%, with most of the values being closer to the latter.

Table 1
Analyses and characteristics of clinkers used

	Clinker no.					
	1	2	3	4	5	6
% SiO_2	22.53	21.10	21.18	23.18	19.47	22.31
% Al_2O_3	5.66	5.15	5.89	2.66	6.37	4.80
% Fe_2O_3	2.84	3.86	2.56	4.21	2.21	3.26
% CaO	63.68	67.70	64.25	65.61	64.52	63.76
% MgO	3.28	1.23	1.97	2.63	2.74	1.00
% SO_3	0.38	0.07	2.27	0.74	1.86	2.48
% Na_2O	0.36	0.11	0.38	0.14	0.32	0.29
% K_2O	0.93	0.18	0.96	0.61	1.27	0.92
% TiO_2	0.30	0.31	0.30	0.14	0.26	0.15
% P_2O_5	0.09	0.30	0.14	0.04	0.34	0.19
% Mn_2O_3	0.06	0.02	0.22	0.06	0.07	0.13
% SrO	0.03	0.08	0.07	0.05	0.25	0.19
% Total	100.14	100.11	100.19	100.07	99.68	99.48
% Na_2O_{eq}	0.97	0.22	1.01	0.54	1.15	0.90
Optimum % SO_3	3.69	2.78	3.97	3.48	3.89	3.89
% C_3S	43.31	71.03	54.41	65.83	64.70	50.82
% C_2S	32.00	6.99	19.76	16.87	7.09	25.70
% C_3A	11.23	8.73	12.44	0.40	14.73	8.11
% C_4AF	8.64	11.75	7.79	12.81	6.73	9.92

The next step was to prepare the cements actually used in this study. For each of the clinkers, three separate cements of different sulfate contents were formulated by blending different amounts of Terra Alba gypsum with the clinker. Specifically, cements were prepared by adding enough gypsum to each clinker (1) to reach an SO_3 level 1% less than its optimum SO_3 value, (2) to reach its optimum value, and (3) to reach 1% more than its optimum value. Thus, the effects of the magnitude of the gypsum addition to each clinker on the properties of the resulting cement could be assessed over a range of clinkers of varying sulfate "demand".

In producing these cements, the previously ground gypsum was effectively blended with the ground clinker by manual mixing in a mixing bowl for several minutes, followed by 30 min of vigorous agitation in a paint shaker, and, finally, by several additional minutes of manual mixing.

The final suite of cements thus produced consisted of 18 individual cements, based on six widely varying clinkers, each of which was blended with three designated levels of gypsum. In the nomenclature adopted here, each cement is identified by clinker number and a suffix letter: "A" for sulfate content 1% less than optimum, "B" for optimum sulfate content, and "C" for sulfate content 1% more than optimum.

3. Mortar preparation, heat treatment, and subsequent exposure

Mortars were prepared from each of the cements described above at a single constant water/cement ratio of 0.44 and a single constant sand/cement ratio of 2.5. Deionized water was used in all mixes.

The sand used conformed to ASTM C 778 and was derived from the Ottawa, IL deposit (the well-known "Ottawa sand"). To get an appropriate particle size distribution for mortar preparation, a proportion of 60% by weight of the normal ASTM C 778 graded sand was blended with 40% by weight of "20–30" Ottawa sand, i.e., sand sized to pass the No. 20 sieve and retained on the No. 30 sieve.

The choice of sand for DEF experiments may be of some importance. The Ottawa sand used here was tested for potential alkali reactivity (at 80 °C) using ASTM C 1260, and the results indicated nonreactivity. On the other hand, samples of eight locally available sands and a sample of German standard sand (DIN EN 196-1) were also tested in accordance with this test procedure, and all, including the DIN sand, showed relatively high expansion levels (between 0.10% and 0.20% at 16 days after casting), thus indicating potential for ASR reactivity.

The mixing and casting of mortar specimens was patterned after that specified in ASTM C 109. Two types of mortar specimens were prepared: standard expansion bars $25 \times 25 \times 280$ mm in size with embedded length measurement studs and 51-mm cubes designed for strength testing. All of the mixing and specimen fabrication processes were carried out at 23 °C. After mixing, mortar specimens used in this experiment were exposed to three different curing regimes: continuous curing at room temperature (23 °C), and early heating to 55 and 85 °C maximum temperatures before subsequent moist room (and room temperature) exposure. In discussing the results later in this paper, the designation "rm" is used to identify samples continuously cured at room temperature while, the designations "55" and "85" were used for samples exposed to maximum early heating temperatures of 55 and 85 °C, respectively.

As is usual in DEF laboratory experiments, heat treatment was carried out in such a way as to somewhat reflect commercial steam-curing practice. Specimens designated for heat treatment were precured in their molds at 23 °C for 5 h prior to heat treatment. One set was heated to 55 °C; another to 85 °C. The heating rates for the 55 and 85 °C cycles were respectively 16 and 21 °C/h, and in both cases, the maximum temperatures were maintained for 6 h. Cooling rates were approximately similar to the heating rates.

The conditions of exposure of the specimens while being heated may be of importance. In the present work, specimens were precured for 5 h in their molds that were stored in plastic boxes (with lids) that were kept in a fog room. After the 5 h of precuring, the specimens, still in their molds, were placed in a large plastic bag, with about 1 or 2 kg of water at the bottom, and were sealed. The bags were then placed in water in a large container, and the whole unit was then placed in an oven for heat curing. After the heating period, the assemblages were left undisturbed while cooling, and for the remainder of the initial 24 h period, and were then demolded.

A companion set of specimens was prepared in a similar manner but was cured at 23 °C before demolding at 24 h and subsequent exposure.

In DEF studies, the conditions of long-term exposure of mortar specimens may also be of importance. In many studies, specimens are simply placed in water, often with the volume of water unstated or uncontrolled. It is obvious that thin specimens treated in this fashion can undergo extensive leaching, and alkalis, OH^- ions, and perhaps sulfate ions from the pore solutions within the mortars are leached into the surrounding water, thus obviously affecting the results.

In the present study, the mortar bar specimens were supported a few centimeters above a free water surface within "shoe-box" type plastic containers, maintained within a fog room. Unfortunately, the development of small temperature differences resulted in the condensation of water on the upper surfaces of the specimens. This condensed water percolates through the specimens, carrying ions from the pore solutions into the free water reservoir. Thus, this form of long-term exposure is also subject to uncontrolled removal of components from the mortar specimens.

Specimens were maintained in this environment for a period of up to 900 days after heating, and continue to be monitored.

In addition to the mortar bar specimens described above, 51-mm mortar cube specimens were prepared from each mortar mix. Replicate mortar cubes were subject to each of the treatments that the mortar bars were exposed to, i.e., heating to 85 °C, to 55 °C, and to continuous room temperature exposure. After the first day, the mortar cubes were demolded and positioned over a free water surface in plastic boxes in the same manner as described for the mortar bars. However, the level of water in the storage boxes in which some of the mortar cube specimens were confined gradually rose during the exposure period due to condensation, and toward the end of the exposure the bottoms of several of the test mortar cubes were actually immersed in water, to a depth of several millimeters. These mortar cubes were designed for measurements of compressive strength, the results of which will be reported in a subsequent paper.

In all, 54 separate mortar series (6 clinker × 3 sulfate levels × 3 early curing temperatures) were evaluated in this study.

4. Procedures used for measurements of expansion, dynamic modulus of elasticity, and weight gain

A variety of experimental measurements and observations were conducted periodically on the mortar specimens described above. In the present paper, we describe the results of three significant measurements: the linear expansion, the dynamic modulus of elasticity, and the weight gain. Subsequent papers will describe the results of quantitative determinations of the amount of ettringite formed under various conditions and will document microstructural changes observed in the expanding mortars.

Length measurements using a standard dial gage apparatus were carried out at 23 °C at ages of 1, 3, 7, 14, 28, and 56 days, and subsequently at approximately 50-day intervals. The length comparator used met the requirements of ASTM C 490, and the results reported are averages for two mortar bar specimens. The percent expansions were calculated with respect to the base measurement secured at 1 day.

The dynamic modulus of elasticity was measured on the same mortar bars, and these determinations were carried out in conformity with ASTM E 1876. The measurement involved the determination of the density of the specimen (after removing excess water) and then of the fundamental transverse resonance frequency of the bar. This frequency was determined using a Grindo-Sonic MK4X instrument that operates in the impact excitation mode; the bar is supported as a simple beam, struck with a small impactor, and the fundamental transverse resonant frequency is determined electronically. The procedure consisted of exciting the mortar bar by a simple elastic strike and picking up the resulting mechanical vibrations with a piezolelectric transducer. The instrument transforms the collected vibrations into electric signals. The signal analyzer isolates the fundamental resonant frequency, which is then displayed numerically on the instrument's panel. The dynamic modulus of elasticity (in psi) is given by the product of three factors: a shape-related coefficient; the weight of the specimen; and the square of the fundamental transverse frequency. The shape-related coefficient is calculated from the dimensions of the specimen and its estimated Poisson's ratio, as provided in the ASTM C 215 standard procedure.

Finally, the surface dry weight of the specimen, measured in order to determine the density of the specimen, itself provided an independent datum linked to the onset and progression of the DEF process.

5. Results: visual observations

Mortar bar specimens were observed periodically as part of the process of carrying out the measurements to be described below.

No significant changes were observed for the many mortar bars that did not experience expansions. However, those that did eventually became badly cracked and in many cases were noticeably warped. It was observed that the top surfaces of the affected mortar bars were much more extensively cracked than the bottom surfaces. The direction of curvature indicated that the upper part of the specimens underwent greater expansion than the lower part, possibly due to frictional restraint exerted by the supports on which the lower surfaces of the mortar bars rested. It is also possible that the larger expansions noted for the top of the bars is related to the presence of condensed water on the upper surfaces. It was also observed that in many of the bars, the end portions (which contained the steel studs) were more severely cracked than the middle portions.

The mortar cubes showed a very different pattern of behavior. There was no visible damage on the upper surfaces or the side surfaces of any of the cubes. They also did not show any indication of warping or expansion. Mortar cubes made from the mortars that exhibited severe expansion as bar specimens showed only slight cracking on (or close to) the bottom surfaces that had been partly immersed in water.

6. Test results

6.1. Expansion

In discussing these results, it is obvious that a consistent and easily recalled nomenclature is needed to facilitate the comparisons. Accordingly, each mortar series is described by a three-component code designation: the number of the clinker, as indicated in Table 1, followed by the letter A, B, or C, reflecting SO_3 contents 1% less than optimum, optimum, or 1% more than optimum, and, finally, a third element designating the exposure history of the mortar.

Comparisons with respect to expansion are greatly facilitated by the fact that only 6 of the 54 mortar bars types actually experienced expansion. Of these, four represented "C85" mortars from four different clinkers. The other two, showing much less expansions, were "B85". None of the lower-than-optimum SO_3 content mortars exhibited any expansion. Similarly, no expansion was observed for any of the mortars cured continuously at room temperature, or any of the mortars heated to 55 °C.

Fig. 1 provides the expansion vs. time data for the six mortars that experienced expansion.

As can be seen in Fig. 1, the onset of expansion varied from about 250 days (about 8 months) to perhaps twice this: it is very difficult to delineate the actual onset of the gradual expansion exhibited by the two "B85" mortars. However, for most expanding specimens, the onset of expansion was marked by a number of changes in both the microstructure and the macroscopic appearance of the affected specimens. Discussion of these changes will be presented in a subsequent publication.

The magnitudes of the expansions after ~900 days of exposure was about 1.7% in one case (5C85)—which appears to have stabilized—but were less than 1% for all of the other expanding mortars, all of which appear to still be expanding.

6.2. Dynamic modulus of elasticity

The normal range of values of the dynamic elastic modulus of mortars that are not undergoing DEF or other durability problems is of the order of 34.5–41.3 GPa. The usual time pattern observed is a rapid increase followed by a very slow increase with time, extending over several years. The slowly increasing values are usually

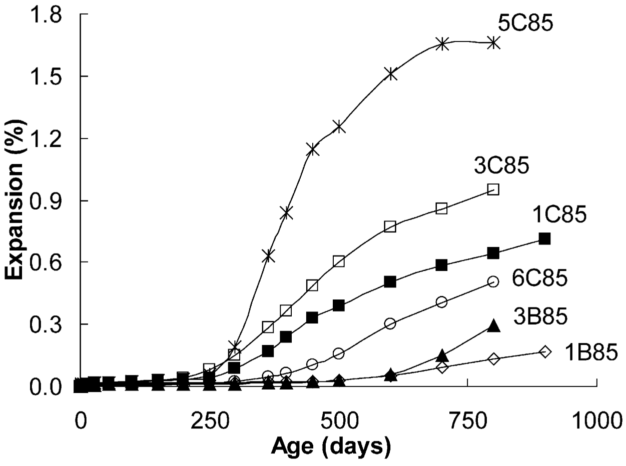

Fig. 1. Expansion–time plots for all 85 °C cured mortars experiencing expansions.

considered to reflect the effects of small increases in the degree of hydration.

This time pattern was found for all of the nonexpanding mortars in the present study. Examples are illustrated in Fig. 2a, which shows the elastic modulus response for the 6B series of mortars cured in three different conditions. None of these mortars expanded. It is seen that, initially, the elastic modulus increases rapidly to the level of about 38 GPa, and then undergoes modest but progressive further increases with time. Small differences occur and persist between the mortar specimens exposed to the different curing conditions.

Fig. 2b provides corresponding results for mortars prepared from the 6C cement, i.e., the cement containing an SO_3 content 1% above optimum derived from the same clinker. The patterns for the entirely room-temperature-cured mortar and for the 55 °C mortar are similar to those in Fig. 2a. In contrast, for the 85 °C mortar, the only one in the set that showed expansion due to elevated temperature exposure, the dynamic modulus of elasticity leveled off at about 300 days and then decreased. The decrease was gradual at first, and then it became more pronounced. By 700 days, the value had dropped to about 34.5 GPa, i.e., a reduction from the maximum value of a little more than 10% was observed.

Fig. 2c shows the dynamic modulus changes with time for two mortars made using Clinker No. 5: one for the 5Brm mortar that did not expand and the other for the 5C85 mortar that exhibited the highest expansion of all the mortars examined. For the latter, the percentage reduction in elastic modulus from the peak value reached was almost 40%, the modulus dropping to a low value of about 24 GPa at 750 days.

In general, it was found that a significant reduction in dynamic modulus occurred for five of the six specific mortars that experienced expansion. The sixth, 1B85, exhibited long-delayed and only very modest expansion (0.2% by 900 days). Its elastic modulus peaked at about 250 days, but then it remained virtually constant, showing only a slight, numerically insignificant reduction. In contrast, all of the remaining 48 mortars that did not expand displayed slow,

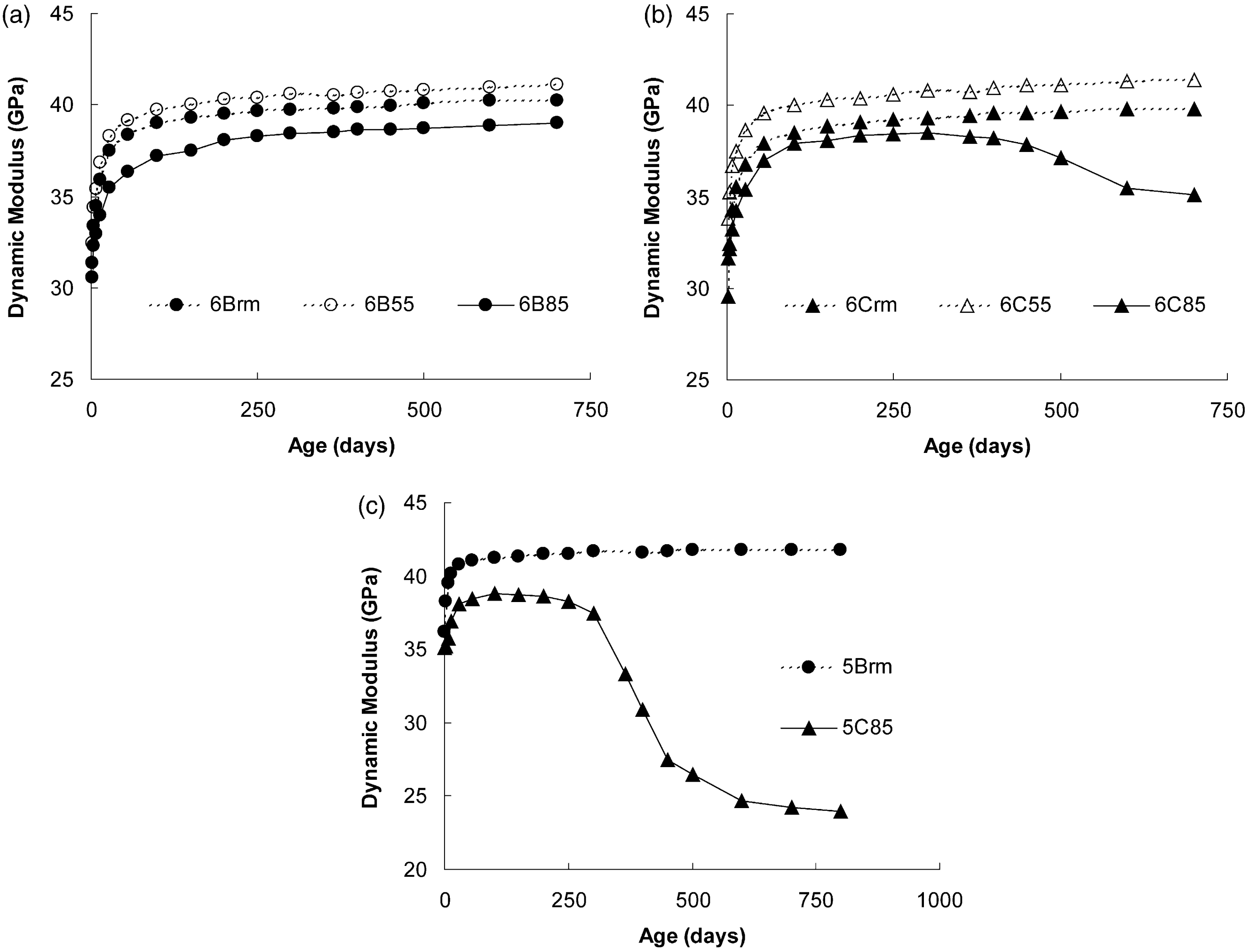

Fig. 2. (a) Dynamic modulus of elasticity vs. time curves for 6B mortars exposed to different temperature-curing regimes. None of these mortars showed expansion. (b) Dynamic modulus of elasticity vs. time curves for 6C mortars. Only the mortar specimens exposed to 85 °C expanded. (c) Dynamic modulus of elasticity vs. time curves for two mortars prepared from Clinker No. 5: 5Brm and 5C85.

progressive increases in elastic modulus with time, as illustrated for the nonexpanding mortars in Fig. 2a, b, and c.

Comparison of the time at which the elastic modulus leveled off and started to decrease for expanding mortars with the time at which the expansion accelerated suggests that the two phenomena are closely related and are merely different manifestations of the same process.

It is furthermore evident that the magnitude of expansion is closely associated with the magnitude of the reduction in dynamic elastic modulus. In Fig. 3, we provide the percent reduction in elastic modulus at 700 days (from the maximum value reached earlier) vs. the degree of expansion exhibited at that time. The proportional relationship between the magnitudes of the two effects is evident. Furthermore, it can be seen that the reduction in dynamic elastic modulus did not take place until the magnitude of expansion exceeded about 0.13%.

Reduction in dynamic elastic modulus in cement-based materials is usually taken as an indication of the development of microcracking as well as visible cracking. Detailed examinations of these mortars by backscatter-mode SEM, to be described in a subsequent publication, confirms that this is indeed the case with the present mortar bars.

6.3. Weight gain

It has been previously indicated that the density of the mortar bar specimen is an element in the calculation of the dynamic elastic modulus by resonant frequency measurements. In order to facilitate the necessary density calculations, weight measurements were taken on all specimens as part of the routine of elastic modulus determinations. Specimens were blotted surface dry (wiped with absorbent cloth until all visible films of water were removed) at the time of the weight measurement, but not otherwise disturbed.

It was found that mortars that did not undergo expansions and the concomitant reductions in dynamic elastic modulus followed a common weight gain–time pattern. They typically showed rapid increases in weight (on the order of about 1%) over the first 28 days; between 28 and 250 days, progressively smaller additional increments were noted; and after about 250 days, the weights stabilized. The steady-state weight levels were about 1% to about 2% higher than the 1-day base weights.

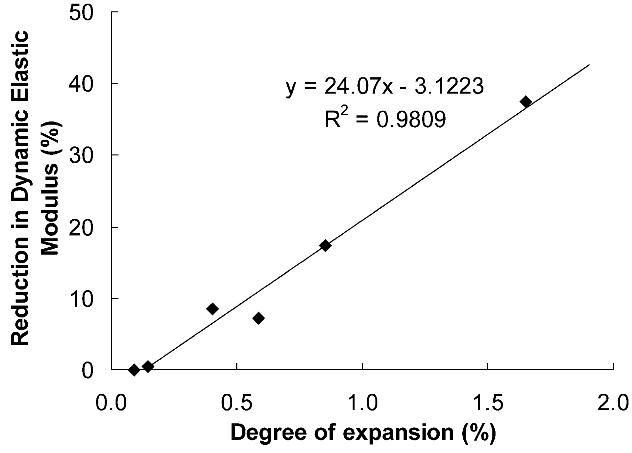

Fig. 3. Expansion vs. % reduction in dynamic modulus of elasticity at 700 days.

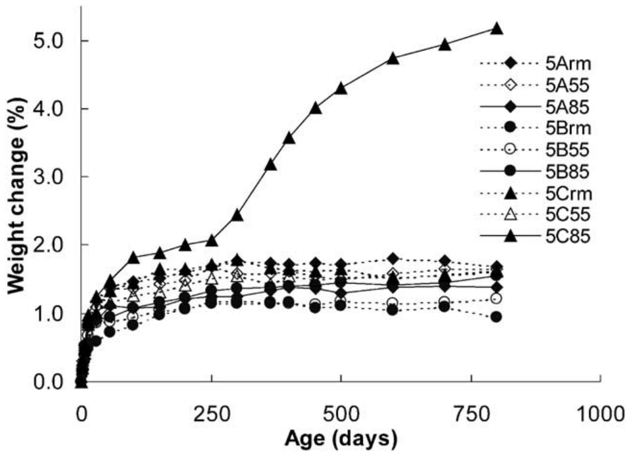

Fig. 4. Weight changes of all mortars specimens made with cement 5.

In contrast, it was found that mortars undergoing expansions and concomitant reductions in dynamic elastic modulus showed a rather different history. For these mortars, increases in weight in the early periods were typically larger than for mortars not experiencing the expansion and the specimens did not reach a constant weight level. Instead, these samples experienced an accelerated gain in weight, especially after the onset of expansion.

Fig. 4 illustrates this behavior for the full set of mortars prepared from Clinker No. 5. Of these, only the mortar designated as 5C85 exhibited expansion; the others did not. The difference in weight gain behavior of the expanded mortar from those of the others is obvious.

In Fig. 5, we compare the weight gain characteristics for the six mortars exhibiting expansions as previously illus-

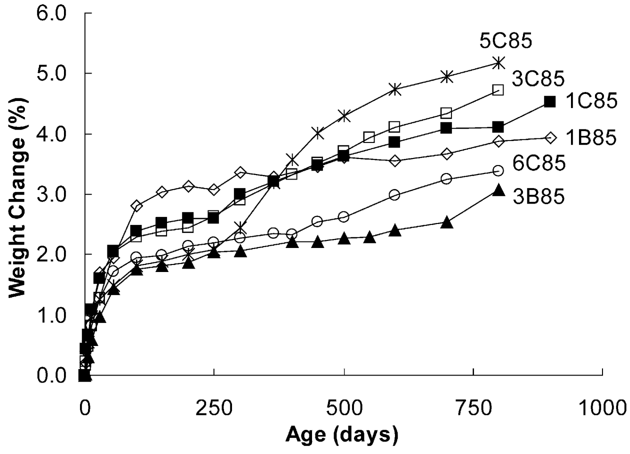

Fig. 5. Weight gain–time plots for all mortar specimens exhibiting expansion.

trated in Fig. 1. It is seen that the weight gain–time patterns for the expanding mortars are different from those found for nonexpanding mortars, as shown in Fig. 4. However, each of them differs in detail from one another. The 5C85 mortar shows a very distinct two-step pattern, with a temporary halt in weight gain separating two distinct weight gain periods. The 6C85 and 1C85 mortars both show an indication of similar stepwise weight gain (the separation between the steps is not so clearly exhibited, however), but the others do not. The 3B85 mortar shows a significant weight increase step only after 700 days, i.e., only as the very much delayed expansion take place.

Comparison of Fig. 1 with Fig. 5 suggests that there is some relationship between expansion and weight gain, with the step increment in weight gain commencing at the time when expansion first becomes noticeable. However, the two phenomena are not completely correlated. For example, mortar 5C85 shows almost twice the expansion of the mortar showing the next-highest expansion, 3C85, but the weight gains of the two are quite similar in magnitude. In addition, mortar 1B85 shows higher weight gain but less expansion than the 3B85 mortar.

7. Discussion

A number of points are evident from the experimental observations reported in this paper.

None of the entirely room-temperature-cured mortars and none of the 55 °C heated mortars displayed expansion or cracking. Thus, within the compositional range of the cements examined, heating in excess of 55 °C is necessary to develop the expansion levels similar to those exhibited by 85 °C heated mortar bars used in this study.

Within the set of 85 °C heated mortars, SO_3 contents higher than the optimum sulfate for the particular clinker are associated with early expansion. The four cements that produced significant expansions prior to about 400 days were all “C” cements, with actual SO_3 levels ranging from 4.7% to 5.0%. Of the two “C” cements that did not produce expansion, one was made from a clinker that had an extremely low alkali content ($\sim$0.2% $Na_2O_{equiv.}$), and the other had an extremely low Bogue C_3A content (only 0.4%).

However, an SO_3 content significantly higher than optimum does not seem to be an absolute necessity for some expansion to manifest itself. For example, mortars made from two of the six “B” cements (1B85 and 3B85) heated to 85 °C did show some evidence of expansion, but the expansions observed were much lower than those observed in the corresponding “C” cement mortars and were significantly delayed.

At the time when this paper was written, the mortars were stored for about 750–800 days and were still expanding. Since than, the mortars have been stored for an additional 1000 days, thus accumulating a total expansion time between 1750 and 1800 days. The expansion rates slowed down significantly and seem to have reached a plateau. However, the previously described trends still exist, that is, the expansion of B85-series mortars is still lower than the expansion of C85-series mortars.

The weight gain effect recorded here is of considerable interest. It should be noted that the numerical percentages of increase reported reflect changes in *mortar* weight. Calculation indicates that a 1% increase in mortar weight would correspond to an almost 4% weight increase per unit weight of *cement* (0.01 mortar weight increase/0.25 unit weight of cement in mortar = 0.04), assuming as a base the combined weights of cement, sand, and water originally mixed. The increases are measured with respect to the first weight measurement at 1 day.

A weight gain effect associated with DEF expansion was previously noted, for example, by Grattan-Bellew et al. [8] and Lewis [6], for mortar specimens immersed in water. They reported that the weight gained was proportional to expansion, which is not true in the present case, and indicated that it was likely associated with the formation of ettringite. The present authors are of the opinion that the observed weight changes might be due to a combined effect of ettringite formation and trapping of the water in the cracks of the mortar that form during the expansion process. However, further investigation is needed to confirm that theory.

The difference in the DEF response of the same mortar exposed as thin mortar bars and as mortar cubes appears to reflect differences in the extent to which alkali is removed from the pore solution of the mortar by progressive leaching. As indicated by Famy [9] and Diamond [10], the onset of DEF symptoms appears to require prior removal of most of the alkali hydroxide contained in the pore solution; this is not readily accomplished with mortar cube specimens.

8. Conclusions

(1) Within a suite of cements prepared to contain SO_3 levels 1% less than optimum, optimum levels, and levels 1% greater than optimum, extensive DEF-induced expansion was produced only in high-temperature (85 °C)-treated mortars made from the “oversulfated” set. Two mortars containing the optimum SO_3 content cements also showed modest expansions. None of the mortars made from cements with SO_3 contents less than optimum showed any evidence of DEF. It is obvious that high SO_3 content in cement is a significant factor in DEF. Although the 5B85 specimen did not show expansion at the time when this paper was written (at that time they were stored for about 750 days), they started expanding after about 1000 days of storage and after 1750 days of exposure reached an expansion level of 0.3%. It should be noted that an optimum sulfate content for this series (3.89%) was similar to that of the 1B85 series (3.69%), which started showing expansion at about 500 days. On the other hand, the specimens from the 2C85 series, for which

the optimum sulfate content was lower (2.78%), did not show any expansion after 1800 days of exposure.

(2) High-temperature-treated mortars made from two of the six "oversulfated" cements failed to expand. Of these, one had an extremely low alkali content, the other an extremely low C_3A content. Presumably, high alkali contents and appreciable C_3A contents are required for DEF to be developed.

(3) No expansions or other symptoms of DEF were developed in mortars that were exposed to 55 °C treatment or to continuous room temperature curing.

(4) In each case, DEF-induced expansions were accompanied by significant and progressive reductions in dynamic elastic modulus, attributed to the progressive crack development within the affected mortars.

(5) In each case, DEF-induced expansions were accompanied by significant increases in the weight of surface-dry specimens, over and above the more modest weight increases exhibited by companion specimens not showing DEF symptoms.

Acknowledgements

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