Effect of Curing Temperature and Cement Chemistry on the Potential for Concrete Expansion Due to DEF*

by Sharon L. Tracy, Stephen R. Boyd, and James D. Connolly

Comments by Paul D. Tennis, Richard W. Burrows, and Authors

PAUL D. TENNIS†

I read this article with much interest. The authors' recommendations on control of curing temperature appear to be the best advice, in light of the current state of knowledge about the DEF (delayed ettringite formation) phenomena. It is interesting to note that in Phase II of the research, 24-hour strengths showed no improvement with increased curing temperature beyond 65°C in almost every case [see Table 8 (p. 50) in the article].

However, I am concerned with the suggested "threshold limits" based on the "Δ ettringite" value for several reasons. First, the reproducibility of the value produced by the procedure appears to be very high. Table 9 [9.51] in the article provides five instances of duplicate values for test results: each cement was tested at 70°C twice. The difference in these duplicate test values vary from 0 to 100 percent and averages more than 50 percent.

Further, in four out of five of these cases, the results would place the cements in different "risk" categories depending on which test result was considered. This appears to indicate that the test reproducibility is not sufficient to establish the "threshold limits" suggested in the article. Logically, I am not sure that there is any reason to expect that the forced hydration procedure would have any reason to mimic real-world concrete behavior or that ettringite forming in this procedure beyond 24 hours would necessarily be deleterious.

I believe the article is valuable in that it provides additional data on a complex topic, and may stimulate further research in this area. However, I believe the authors' implied recommendations on using the results of this experimental test procedure went beyond that which could be justified by their data.

* PCI JOURNAL, V. 49, No. 1, January-February 2004, pp. 46-57.

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RICHARD W. BURROWS‡

The authors and Wiss, Janney, Elstner, Associates are admired for their commitment to investigations that take years to complete. Their investigation involved a tremendous amount of work and is insightful, valuable and very well presented. Their findings are consistent with those of Heinz et al.[1] who showed that, in tests of 90 cements, whether or not a cement would suffer from DEF (delayed ettringite formation) depended on the ratio of SO_3 to Al_2O_3; cements with a ratio exceeding 0.45 were shown to be vulnerable.

Fig. A is a plot of the Heinz data on which the WJE data has been superimposed. The correlation is quite good, as Cement No. 5 did not expand and Cements 3 and 4 had the worst expansions. The correlation would have been perfect if Cement No. 1 had been displaced a little to the right.

It was interesting that Cement No. 5 did not expand even when heat cured at 90°C and that, at a temperature of 65°C, only one cement, Cement No. 4, expanded – and that expansion began only after two years of moist storage. DEF requires a long exposure to moist conditions. This information is valuable for understanding the deterioration of some box beams in Texas where the experts were divided on whether the cause was DEF or ASR (alkali-silica reaction). There are divergent schools of thought on DEF, perhaps typified by the theories of Ludwig[1] versus the theories of Mario Collepardi.[2]

Ludwig vs. Collepardi – If vexed by the conflicting morass of information about delayed ettringite formation, one can trust Ludwig who discovered DEF in 1978. His knowledge of DEF and DEF testing procedures have evolved over a period of 25 years and involve heat treating mortar and concrete samples at various temperatures with subsequent long-term moist storage without any drying of the samples.

Mario Collepardi has a completely different view, claiming that the essential requirements for DEF are pre-existing microcracks plus cycles of wetting and drying. He suggests that ettringite forms at the tips of the microcracks and spreads the cracks – causing expansion and destruction.

To support his hypothesis, Collepardi cited the famous case of the railroad ties where the ties in tunnels were not affected while the ties on the top of a stored stack (which were wetted by rainfall and then dried) were the first to crack. He also concluded that high temperature (steam) curing was not essential for DEF because some normally-cured ties were also cracking. This is the second conflict as most investigators have only been able to produce DEF by heat curing at temperatures exceeding 65°C.

Adding to this confusion is the case of the cracking box beams in Texas. Some [cement] experts concluded that it (the cause of cracking) was ASR but others (including the Texas DOT) concluded that it was DEF.[3] This impasse I hope to break, by offering a third theory of the deterioration. **It is, I believe, a very simple case of cracking from drying shrinkage caused by a cement with very high levels of both alkalies and fineness.** This theory is based on certain historic events.

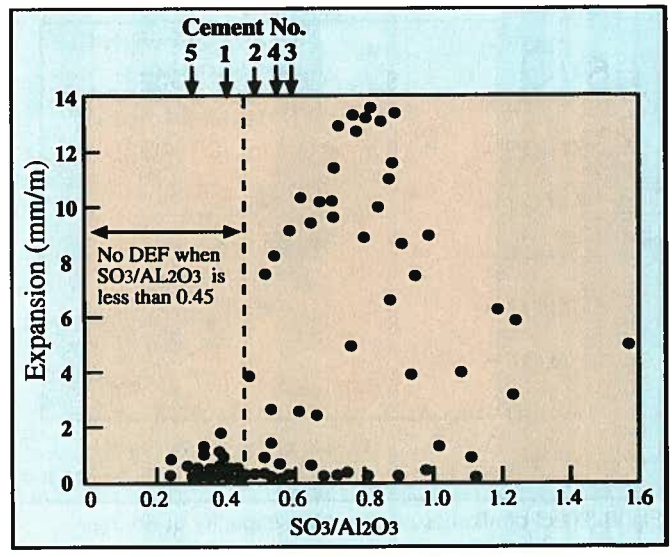

Fig. A. DEF expansion as a function of SO_3/Al_2O_3.

Alkali-associated events from 1929 to 2004 – Cements with alkali contents over 1 percent have a history of causing cracking from drying shrinkage. This fact gets discovered, forgotten, rediscovered and forgotten again ever since the time of Thaddeus Merriman. As a result, the cracking is often misdiagnosed as alkali-silica reaction (ASR) or delayed ettringite expansion (DEF).

Thaddeus Merriman (1929) – Merriman, who was chief engineer for the New York City Water Board, gave a paper in Tokyo in 1929.[4] He stated that the alkalies in cement had to be controlled to ensure orderly hydration. He solved a drying shrinkage cracking problem by washing the alkalies out of the cement.[5] Two years before ASR was discovered at Parker Dam, he specified a low-alkali cement for the Water Board – a cement still being produced by Blue Circle as "Merriman" cement.

Harmon Meissner (1942) – The cracking at Parker Dam was investigated in 1942 by Harmon Meissner of the U.S. Bureau of Reclamation (USBR).[6] He described it as drying shrinkage cracks which were being widened by the ASR expansion of the concrete 2 ft below the surface. Then everybody promptly forgot about the "drying shrinkage" part of the equation - except Meissner. He made test slabs of low and high-alkali cement and reactive and non-reactive aggregates.

As expected, the slab with the high-alkali cement and the reactive aggregate cracked. What was not expected was that the slab with the high-alkali cement and the non-reactive aggregate also cracked, but not as badly. As a result, the Bureau specified that low-alkali cement be used regardless of the aggregate. This policy is still in effect today although its origin has probably been forgotten. In another USBR investigation, a test slab with high-alkali cement and a non-reactive aggregate was observed to crack. It was thought that a batching error had been made. A new slab was cast. It also cracked.

R. L. Blaine (1969) – The issue surfaced again when R. L. Blaine, of the National Bureau of Standards, tested 199

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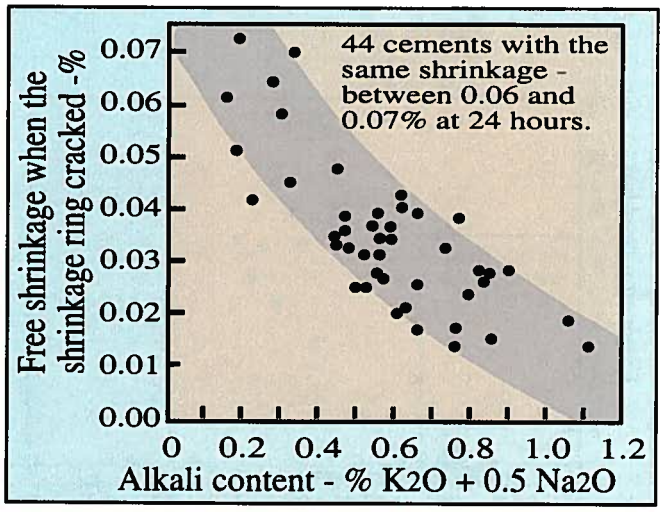

Fig. B. Effect of alkalies on the strain capacity of drying cement pastes of the same shrinkage.

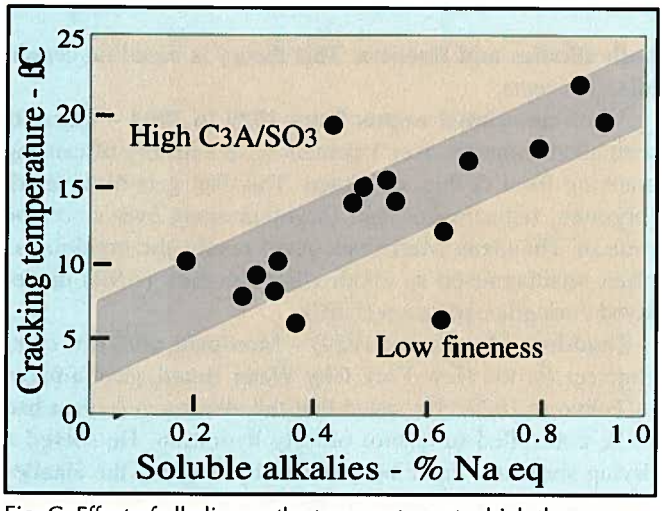

Fig. C. Effect of alkalies on the temperature at which the concrete cracked as it cooled down from the heat of hydration.

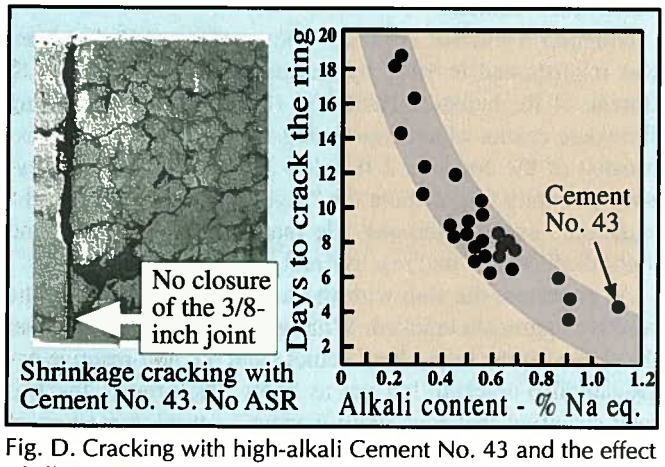

Fig. D. Cracking with high-alkali Cement No. 43 and the effect of alkalies on shrinkage cracking.

cements between 1953 and 1965.[7] He used the ring shrinkage test to study the cracking from drying shrinkage and found that the two greatest factors were the alkali content (especially the potassium ion) and the fineness. His test was considered for a RILEM test but the cement people said that tests on cement paste were not relevant to concrete and so the issue died once again.

Fig. B shows Blaine's results on the effect of alkalies on the strain capacity of cement paste. Forty-four cements with the same free shrinkage demonstrated widely different strain capacities depending on the alkali content. Note that the strain capacity of the 1.2 percent K_2O cement was only about one-seventh that of the lowest alkali cements.

Mori (1974) **and Ramchandrun** (1993) – Examination of hydrating cement pastes conducted with the SEM by Mori et al. in 1974[8] and later by Ramchandrun et al. in 1993,[9] revealed that the alkalies cause the tricalcium silicate to hydrate as rod-like structures instead of the desirable acicular (needle-like) structures. This is probably the cause of the marked reduction in the strain capacity that Blaine found. It is also the probable cause of the reduced 28-day strength with high-alkali cement. The early strength is increased because alkalies accelerate the rate of hydration. This increases the thermal cracking of bridge decks.

R. E. Oberholster et al. (1983) – In 1983, events such as those described below caused R. E. Oberholster in South Africa to write that ASR should never be diagnosed unless there is positive evidence of expansion.[10]

Rolf Breitenbucher (1988) – In 1988, Rolf Breitenbucher developed the thermal cracking test now known as RILEM TC 119 TCE 3. He tested many cements and found that the two greatest cement factors in the cracking from thermal contraction were the alkali content and the fineness.[11] These data, shown in Fig. C, parallel Blaine's findings on drying shrinkage. The soluble alkalies are usually about two-thirds of the total alkalies.

Rudolph Springenschmid (1997) – In 1997, a letter was received from Springenschmid that stated that 5 percent of the Autobahn is cracked from the use of a high-alkali cement. The alkali content was about 1.2 percent, Na eq. There was no cracking from ASR. The cracking was due to thermal contraction plus drying shrinkage.

R. W. Burrows (1998) – The author resurrected the alkali issue in 1998.[12] In 1943, the USBR began testing 28 cements in the 104 panels of the parapet wall at Green Mountain Dam as a part of the PCA Long Time Study of Cements. Fifty-five years later, the cracking from drying shrinkage was proportional to the alkali contents of the 28 different cements. This was predictable by Roy Carlson's ring shrinkage test which, in various forms, is now widely used to explore the cracking due to drying shrinkage.

Fig. D shows drying shrinkage cracking with high-alkali Cement No. 43 (1.14 percent Na eq.). This cracking was first misdiagnosed as ASR. However, realistic laboratory tests for ASR showed no expansion, nor was there any expansion at Green Mountain Dam which was measured with an accuracy of 0.001 in. After 55 years, the $^3/_8$-in. contraction joint has still not closed. In Fig. E, the effects of fineness, C_3A and C_3S are also apparent.

Some other events – In 1947, Calvin Oleson observed abnormal cracking of concrete with high-alkali cement in highway structures in Georgia and Alabama.[13] The problem was blamed on ASR even though the aggregate could not be proven to be reactive in laboratory tests. In the laboratory,

deleterious ASR occurred only when 1 percent NaOH was added to a cement with 0.92 percent Na eq. alkalies.

In 1996, Ferrer et al. observed $^3/_8$-in. cracks in a power plant in a hot desert region in Mexico.[14] A high-alkali cement had been used but testing revealed no evidence of ASR. They were drying shrinkage cracks. The alkali content was 1.3 percent.

In 1998, the Lehigh Cement Co. sent the author some photographs of a badly cracked wall in Virginia. It had been diagnosed as ASR but there was no expansion - no squeezing out of the mastic in the contraction joint. They were drying shrinkage cracks.

In 2000, Juenger and Jennings[15] found alkalies increased the cracking from drying shrinkage as did He, Zhou and Li, in 2004.[16] This was not news to the author, although it may have been news to these young researchers not familiar with the above history.

Effect of cement fineness – Fig. F shows the effect of fineness on the cracking time of cement pastes in the ring shrinkage test. Note that the graph does not extend to the 630 m²/kg value of the cement used in the Texas box beams. In Blaine's tests of 199 cements which cracked between four minutes and 45 hours, the worst cement (four minutes) was his finest cement at 590 m²/kg.

Probably, the reason that this cement cracked after only four minutes of drying is that latent stress from autogenous shrinkage was present and the very mild drying of the surface triggered cracking by means of notch sensitivity, a scenario that has been demonstrated by other researchers. There are at least 22 studies in the literature showing that the durability of concrete decreases as the fineness is increased.

Combined effect of high alkalies and high fineness – The U.S. Bureau of Reclamation fabricated concretes with cements of varying finenesses and alkali contents, cured them for 14 days, exposed them to weathering at Denver for a year, and then subjected them to freezing and thawing.[17] The tremendous degree of microcracking with the concretes of high fineness and high alkalies diminished the freezing and thawing resistance from 540 cycles to only 50 cycles as shown in Fig. G. The author, therefore, blames the cracking of the box beams in Texas on the extreme crack-proneness of the cement that was used.

Texas box beams – Lawrence, Myers and Carasquillo[3] have provided a summary of the problem. When the author read that the cement in the cracking box beams had an alkali content of 1.2 percent and a fineness of 630 m²/kg, bells and whistles went off because this was the same alkali content as the cements in the cracking concrete at Green Mountain Dam (1.1 percent), the Mexican power plant (1.3 percent), the Autobahn (1.2 percent), and the railroad ties (> 1.0 percent).

In 1995, the cracking of 56 prestressed concrete box beams that had been in storage for four years initiated intensive investigations. The 56 beams contained a high-alkali Type III cement. Thirteen beams made with a low-alkali Type III cement were not cracking. The experts who concluded that the cause of the cracking was DEF never explained why the beams with the low alkali cement did not crack.

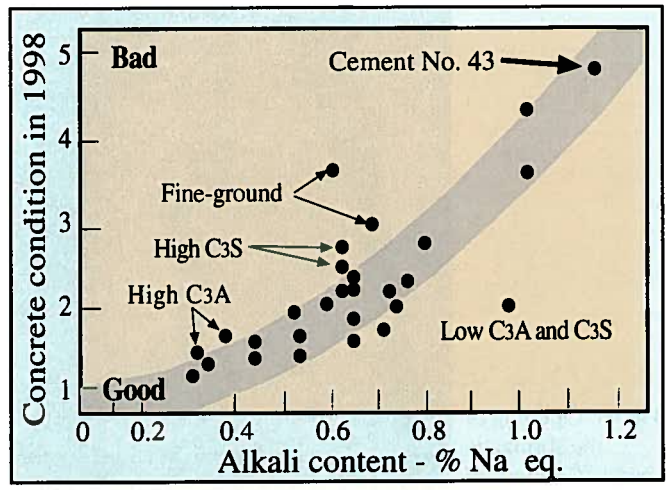

Fig. E. Effect of alkali content on the condition of the concretes after 55 years. There is no ASR .

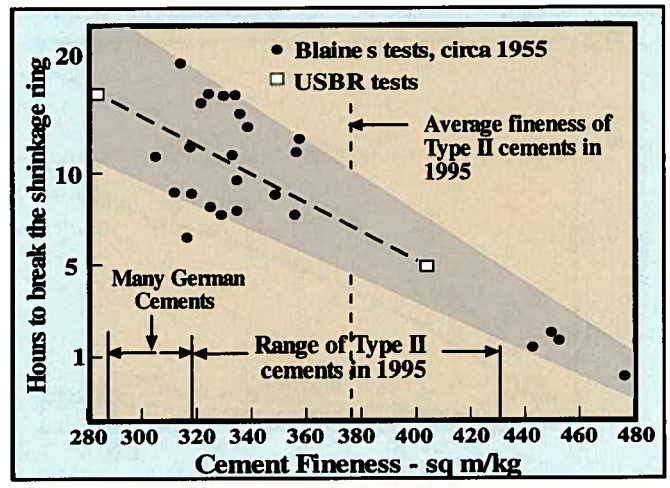

Fig. F. Fineness versus the cracking from drying shrinkage.

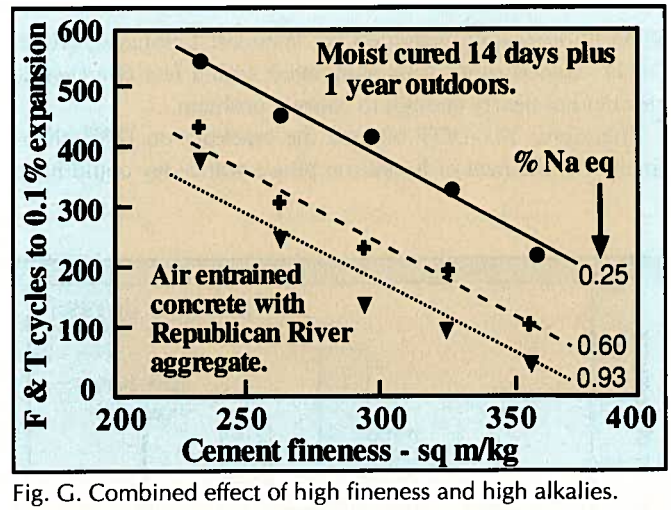

Fig. G. Combined effect of high fineness and high alkalies.

The beams were moist-cured at room temperature. Steam curing was not used. Investigations by the cement supplier blamed ASR, which seemed logical since the beams made with low-alkali cement were not cracking. But the problem is that the aggregate had a good service record. In addition,

Fig. H. Opening of the shrinkage crack. Courtesy of Kevin Pruski, Texas DOT.

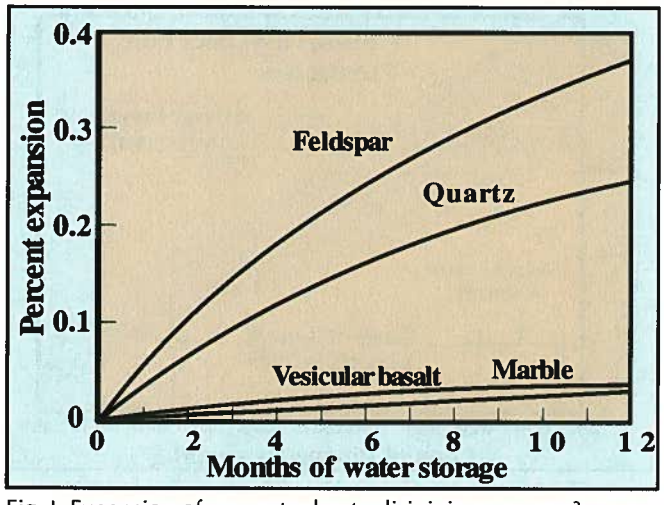

Fig. I. Expansion of concrete due to disjoining pressure?

SEM photographs provided by Wendell Dubberke (Iowa DOT) showed some ASR associated with a few chert particles but not nearly enough to cause a problem.

Therefore, Tex-DOT blamed the cracking on DEF, theorizing that the heat of hydration plus a warm day could have resulted in a temperature of 65°C. However, at this low temperature, several years of moist storage are necessary before expansion even begins and the Texas beams showed cracking in less than a year in a fairly dry environment. Therefore, the author believes that the deterioration was due to the extreme crack-proneness from drying shrinkage that is typical of concrete made with a cement of very high fineness and very high alkali content.

It is theorized that the now wide-gapped longitudinal crack shown in Fig. H began as a small drying shrinkage crack which, through the process of creep and additional shrinkage, opened up as the stressed cables strove to pull the ends together. The beam is buckling and bending which caused the crack to open. The concrete at the far end of the beam has sheared transversely, resulting in a loss of support.

Why is the concrete expanding? – The box beams have been reported to be expanding. This may be due to disjoining pressure. Fig. I shows the expansion of concrete bars in water which were first wet cured 28 days at 70°F, plus 7 days at 130°F, plus 7 days drying at 130°F (12). This produced severe microcracking from drying shrinkage and the bars are expanding for probably the same reason that bentonite clay expands in water-disjoining pressure. Water entering cracks with gaps less than three nanometers create a separating pressure as the water tries to form an adsorbed layer three nanometers thick.

According to Bernal, a pressure of 1000 psi is developed in a one-nanometer crack. Powers wrote[18] that there is experimental evidence that a significant fraction of the internal surface area in cement paste bounds spaces that are too narrow to permit unhindered adsorption. This situation would be aggravated by severe microcracking.

In Fig. I, it is postulated that the stronger paste-to-rock bond with the basalt and the marble was able to resist the disjoining pressure and expansion did not occur. With feldspar, there is neither a chemical nor a physical bond with the cement paste. The author attributes the expansion of the Texas box beams to disjoining pressure. The beams collected rainwater after the crack opened. The Styrofoam core was found to be saturated with water.

Avoidance of cracking problems in the future – In 1995, Ron Gebhardt published his survey of North Ameri-

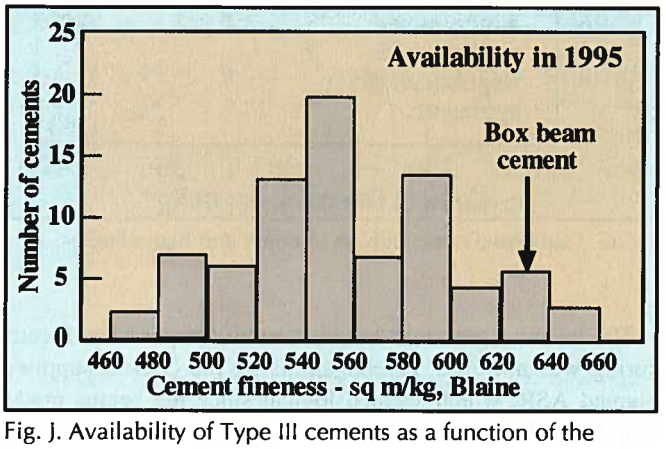

Fig. J. Availability of Type III cements as a function of the cement fineness.

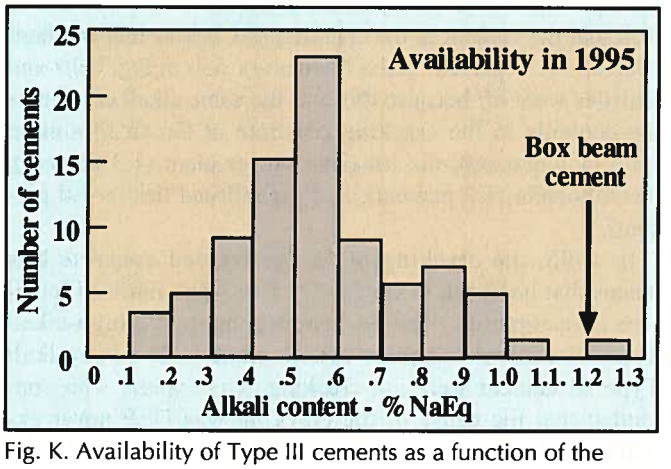

Fig. K. Availability of Type III cements as a function of the alkali content.

can portland cements.[19] Figs. J and K were plotted from Gebhardt's survey. Also plotted is the cement used in the box beams. It had a very high fineness of 630 m^2/kg and a very high alkali content of 1.2 percent.

From the standpoint of the cracking tendency from drying shrinkage and thermal contraction, the high-alkali, high fineness box beam cement was worse than any of the 86 Type III cements surveyed by Gebhardt. The problem could be ascribed to bad luck since it is a matter of chance whether one gets a crack-resistant cement or a crack-prone one. The luck factor can only be eliminated by a tighter specification.

Recommendations

1. In prestressed concrete, avoid cracking from drying shrinkage by specifying a low-alkali Type III cement with a fineness less than 560 m^2/kg. Figs. J and K show that many cements can meet this requirement.

2. To avoid problems from DEF, select a cement with the ratio of SO_3 to Al_2O_3 less than 0.45. This problem is also mitigated by low values of fineness and alkalies.

REFERENCES

1. Heinz, D., Kalde, M., Ludwig, U., and Ruediger, I., "Present State of Investigation of Damaging Late Ettringite Formation (DLEF) in Mortars and Concretes," ACI SP 177-1, *Ettringite, the Sometimes Host of Destruction*, 1999, pp. 1-14.
2. Collepardi, Mario, "Damage by Delayed Ettringite Formation," *Concrete International*, January 1999, pp. 69-74.
3. Lawrence, B. L, Meyers, J. I., and Carrasquillo, R. L., "Premature Concrete Deterioration in Texas Department of Transportation Precast Elements," ACI SP 177-10, Ettringite, *The Sometimes Host of Destruction*, 1999, pp. 141-158.
4. Merriman, M. T., "*Cement,*" Proceedings of the World Engineering Congress, Tokyo, V. 31, 1929, pp. 345.
5. Merriman, M. T., Discussion of a paper by P. H. Bates, "Crazing of Cement Products," Proceedings of the Twenty-First Annual Convention, American Concrete Institute, V. 21, 1925, pp. 131-132.
6. Meissner, H., "Discussion of a Paper by Stanton," *ACI Journal,* Supplement, V. 38, November 1942, pp. 236-242.
7. Blaine, R. L., Ami, H. T., Evans, D. H., Defore, M. R., Clifton, J. R., and Methey, R. G., "Compilation of Data From Laboratory Studies - Part 6," Building Science Series 36, NIST, Washington, DC, V. 42, 1971, 115 pp.
8. Mori, H., Sudoh, G., Minegishi, K., and Ohta, T., "Some Properties of C-S-H Gel Formed by C3S Hydration in the Presence of Alkali," Paper presented at the VI International Congress on the Chemistry of Cement.
9. Ramchandrun, V. S., Beadoin, J. J., Sarker, S. L, and Aimin, X., "Physico-Chemical and Microstructural Investigations of the Effect of NaOH on the Hydration of $3CaSiO_2$," *Il Cemento*, V. 2, 1993, pp. 73-84.
10. Oberholster, R. E., Van Aardt, J. H. P., and Brandt, M. P., "Structure and Performance of Concrete," Applied Science Publishers, New York, NY, 1983, 563 pp.
11. Breitenbucher, R., "Zwangsspannungen und Ribbildung infolge Hydratationswarme," Doctoral Dissertation, Technical University of Munich, Munich, Germany, 1988, 150 pp.
12. Burrows, R. W., "The Visible and Invisible Cracking of Concrete," ACI Monograph No. 11, 1998, 78 pages, 84 graphs.
13. Oleson, C. C., "Abnormal Cracking in Highway Structures in Georgia and Alabama," *ACI Journal*, March 1963, pp. 329-351.
14. Ferrer, M., Camacho, R., and Catalin, M., "A Confusing Case of Alkali-Aggregate Reaction of Concrete Structures in a Desertic Environment," Presented at the ACI (Mather) Symposium, Denver, Colorado, 1996.
15. Juenger, M. G., and Jennings, H. M., "Effects of High Alkalinity on Cement Pastes," To be published in the *ACI Materials Journal.*
16. He, Z., Zhou, X., and Li, Z., "New Experimental Method of Studying Early-Age Cracking of Cement-Based Materials," *ACI Materials Journal*, January-February 2004, pp. 50.
17. Backstrom, J. E., and Burrows, R. W., Discussion of "Observations on the Resistance of Concrete to Freezing and Thawing," by Hubert Woods, *ACI Journal*, V. 27, No. 4, December 1955.
18. Powers, T. C., *The Properties of Fresh Concrete*, published by John Wiley and Sons, Inc., 1968, p. 411.
19. Gebhardt, R. E., "Survey of North American Portland Cements," *Cement, Concrete, and Aggregates*, V. 17, No. 2, December 1995, pp. 145-189.

AUTHORS' CLOSURE BY SHARON L. TRACY,* STEPHEN R. BOYD,† AND JAMES D. CONNOLLY‡

Response to Richard W. Burrows:

The authors would like to thank Mr. Burrows for a very thorough and thoughtful compilation of data regarding the relationship between cement-alkali content, fineness, and cracking. His points are certainly worth consideration when diagnosing concrete cracking problems.

With respect to DEF and his comments regarding our study, it is true that Cement No. 5 was exhibiting the least expansion when cured at 90°C (194°F). However, after the paper had been written, we continued to measure the prisms. At 4$^1/_2$ years of age, the concrete prisms made from Cement No. 5 and cured at 90°C (194°F) started to expand at a relatively rapid rate. These prisms will continue to be monitored. Based on this observation, it appears that if the concrete is cured at a very high temperature such as 90°C (194°F), the risk for DEF is high regardless of the cement chemistry. The rate of expansion and the total expansion due to DEF currently appear to be a function of cement chemistries and curing temperatures at or higher than 65°C (149°F).

As mentioned in the paper, Cement No. 5 had about 3 percent SO_3, low-alkali content (0.5 percent), low SO_3/C_3A (0.3) and SO_3/Al_2O_3 (molar) of 0.7. We agree that SO_3/Al_2O_3 relates to ettringite formation. However, Cement No. 5 has an SO_3 to Al_2O_3 ratio of 0.7 (molar) or 0.56 (weight), but in Fig. A of Mr. Burrows' discussion, he incorrectly plotted our chemical data. The numbers plotted appear to be the SO_3 to C_3A ratio rather than the SO_3 to Al_2O_3 ratio as indicated in this figure. This, in turn, affects his interpretation of the data, and may affect his recommendations at the end of his discussion.

Response to Paul D. Tennis:

It is true that the 24-hour strength results of Phase 2 concrete did not increase with increasing curing temperature between 65°C (149°F) and 80°C (176°F), indicating that increasing curing temperatures above 65°C for strength purposes does not have a good benefit-to-risk ratio.

Regarding Dr. Tennis's concern about the reproducibility of the data, we have several comments. First, the only temperature regime that was duplicated was 70°C (158°F), which is a borderline temperature. Small deviations in temperature around 70°C (158°F) can affect the onset and degree of DEF. Four of the five concrete mixtures that were duplicated did show different *Δ ettringite* values, and one showed the exact same result. The cement that showed the same result is very stable over all temperatures, and only after 5 years is it beginning to expand when cured at 90°C (194°F).

It is important to look at each cement and concrete individually, since the concrete prisms are expanding differently in a manner that correlates to these measured values of *Δ ettringite*. Thus, the differential thermal analysis (DTA) method does appear to be correlating well with performance, even for concrete cured at 70°C (158°F). The following examples describe in detail the relationship between *Δ ettringite* and expansion behavior for all concrete cured at 70°C (158°F), which is plotted in Fig. L:

Cement No. 1 - The Phase 1 value of *Δ ettringite* was 1.8 percent, and the Phase 2 value was 1.2 percent. According to the suggested threshold, the concrete with a *Δ ettringite* value of 1.8 percent is predicted to expand, and the concrete with a *Δ ettringite* value of 1.2 percent may or may not expand. At the time the paper was written, neither of these

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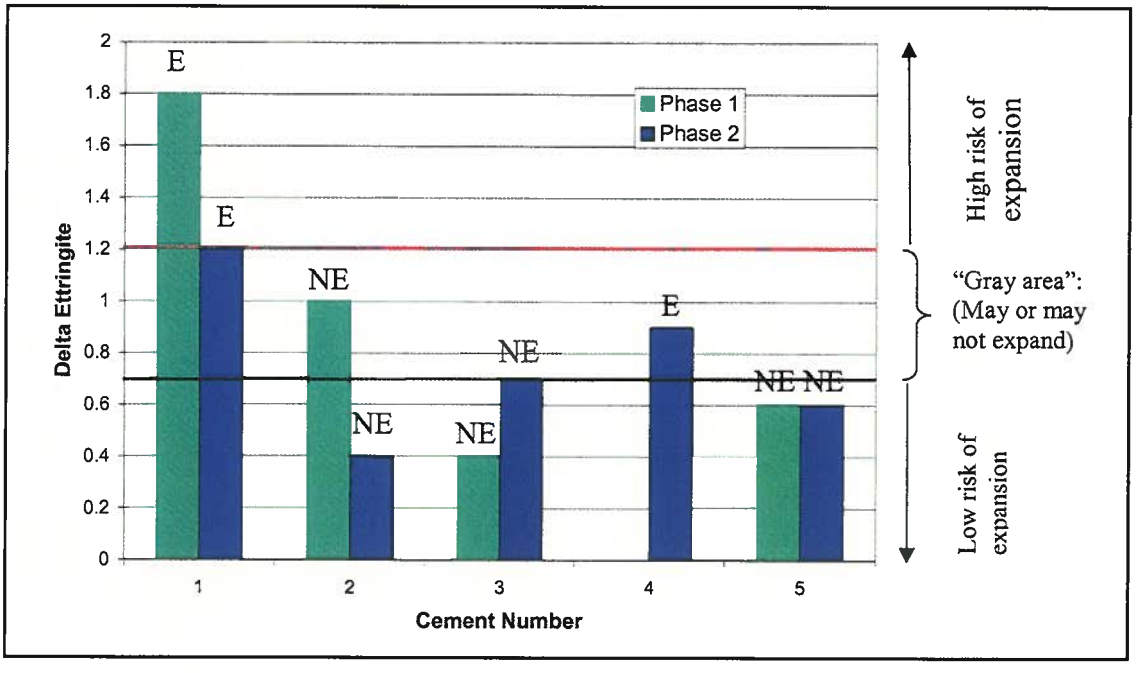

Fig. L. Comparison of *Δ* ettringite results at "borderline temperature" of 70°C (158°F) for both phases of study to suggested threshold values.

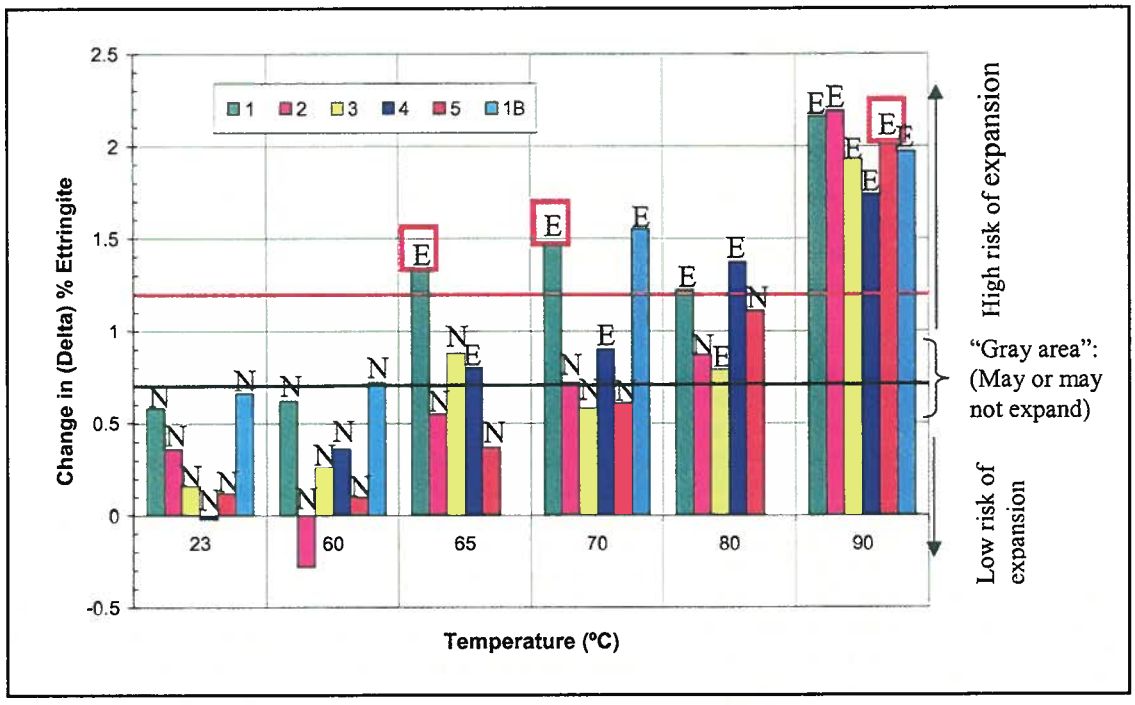

Fig. M. Update of Fig. 11 of the paper. The "E's" in the red boxes highlight concrete that has expanded since the time the paper was written. The red line indicates a suggested new upper threshold, which decreases the "gray area."

concretes was expanding; however, currently both are expanding. The concrete with a *Δ ettringite* value of 1.8 percent has expanded 0.5 percent at 5 years, and the concrete with a *Δ ettringite* value of 1.2 percent has expanded 0.2 percent at 4 years. The predictions hold true.

Cement No. 2 - The Phase 1 value of *Δ ettringite* was 1.0 percent, and the Phase 2 value was 0.4 percent. According to the suggested threshold, the concrete with a *Δ ettringite* value of 1.0 percent may or may not expand, and the concrete with a *Δ ettringite* value of 0.4 percent is not predicted to expand. At the time of the paper and currently neither of these sets of concrete is expanding, and the predictions hold true.

Cement No. 3 - The Phase 1 value of *Δ ettringite* was 0.4 percent, and the Phase 2 value was 0.7 percent. According to the suggested threshold, neither is predicted to expand. Currently, neither is expanding, although all the concrete of this cement cured at 70°C (158°F) or below is showing very slow expansion over time (0.08 to 0.10 percent at 5 years). The predictions hold true.

Cement No. 4 - The background correction for this sample was uncertain in Phase 1 as indicated in the footnote in the paper. The Phase 2 *Δ ettringite* value was 0.9 percent. According to the suggested threshold, a sample with a *Δ ettringite* value 0.9 percent may or may not expand. This sample has expanded 0.8 percent at 4 years. The prediction holds true for the Phase 2 sample.

Cement No. 5 - The same value of *Δ ettringite* was obtained in both phases (0.6 percent). Neither is predicted to expand, and neither is expanding. The predictions hold true.

We are quite pleased with correlation between the predicted expansion based on *Δ ettringite* values versus the actual expansion (currently up to 5 years of measurements). We can even narrow the suggested bands based on the newest data as shown in Fig. M since prisms have begun to expand between the $3^1/_2$ year results reported and 5 years. The width of the uncertain band could be decreased by moving the upper threshold down from 1.4 to 1.2 percent.

In conclusion, the DTA data is internally consistent and correlates to expansion results. The production of DEF is a complicated issue, and many factors are involved. This test may always have a "gray" area where the prediction is not definite of whether a concrete will or will not expand. Further correlations with time will continue to add data to possibly allow the band of uncertainty to be decreased.