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Standard Aggregate Materials for Alkali-Silica Reaction Studies

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ABSTRACT

Preliminary studies have been carried out to identify candidate materials for use as a standard reactive aggregate in alkali-silica investigations. The materials studied included several commercial glasses, an opal, a quartzite, a rhyolite and a calcined flint. Candidate materials were tested according to for their expansion in mortars prepared using either a high-alkali or a low-alkali cement, a nonreactive limestone sand, and some proportion of reactive material. Tests were carried out according to ASTM C 441-81, Standard Test Method for Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction, and ASTM C 227-87, Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method). The proportion of limestone replaced by each reactive material was varied so as to bracket the pessimum proportion (i.e., the proportion of reactive material producing the highest level of expansion). Mortar-bar expansion levels were measured throughout reaction periods of approximately 6 months to 1 year. Expansion results are presented and discussed. With high-alkali cement, the calcined flint produced moderate expansion levels and a rapid early rate of expansion, and with low-alkali cement it produced very little expansion. With high-alkali cement, the Vycor, fused quartz, and fused silica, produced high expansion levels and a rapid early rate of expansion, but with low-alkali cement they produced moderate expansion. Based on these studies, the Vycor, fused quartz, fused silica, and calcined flint appear suitable as standard reactive materials; the calcined flint appears especially promising.

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1. INTRODUCTION

Preventing deterioration of concrete due to chemical reactions between cement and aggregate is a major concern in the production of durable concrete. The principal chemical reaction is the alkali-silica reaction (ASR), which occurs between sodium and potassium, typically derived from the cement, and certain types of siliceous components in the aggregate. The reaction may cause expansion and cracking, and sometimes causes deterioration of the concrete.

Struble [1] recently reviewed the mechanisms of alkali-silica reaction. These mechanisms are understood only in a general sense, and many details of the reaction are still under investigation. Based on an earlier review by Diamond [2,3], the general aspects of the reaction mechanism were summarized as follows:

1. The reaction does not directly involve alkali, but occurs between silica in the aggregate and hydroxyl ions in the pore solution. The concentration of hydroxyl ions is controlled predominantly by the concentration of alkali (sodium and potassium) ions, generally derived from the cement.

2. The product of the reaction is a gel, an amorphous semi-solid or solid material consisting principally of silica, alkali, calcium, and water.

3. The gel may imbibe water and swell, producing expansion and possibly cracking of the mortar or concrete.

A key aspect of the alkali-silica reaction concerns the proportions of alkali and reactive silica in the concrete or mortar. Considerable experience has shown that damage due to alkali-silica reaction does not occur when alkali contents are low. If the cement alkali contents are low, the concentration of hydroxyl ion in the pore solutions are correspondingly low, and the extent of reaction between hydroxyl ion and silica is low. This is the basis for the alkali specification limit of 0.60 percent[1], an optional chemical requirement in ASTM C 150, Standard Specification for Portland Cement. This alkali limit may be invoked to prevent deleterious expansion due to alkali-silica reaction.

The proportion of reactive silica in a mortar or concrete also affects the level of expansion. There is typically a maximum in expansion at some intermediate proportion of reactive

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[1] Expressed as percent of equivalent sodium oxide (Na_2O).

silica, the so-called pessimum proportion. This well-known effect is shown schematically in Fig. 1.

Introducing a substantial proportion of a pozzolanic material, such as fly ash or silica fume, may prevent expansion due to alkali-silica reaction. As described by Diamond [4], these materials appear to prevent expansion by reacting more rapidly than the deleterious aggregate with the alkali solution and producing reaction products that do not themselves swell. To decide whether a specific pozzolanic material prevents expansion, the material may be tested according to ASTM C 441, Standard Test Method for Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction.

To evaluate the effect on expansion of a specific cement or combination of cement and mineral admixture, it is often necessary to measure expansion due to ASR using a standard reactive aggregate. A common standard material is Pyrex glass[2], utilized in ASTM C 441. However, doubts have been raised concerning the future availability of Pyrex glass for this purpose[3], and the performance of the Pyrex glass as a standard reactive material has not always been satisfactory, as discussed later. For these reasons, studies were carried out at the National Institute of Standards and Technology[4] (NIST) to identify candidate materials to replace Pyrex glass in ASTM C 441 and to serve as a standard reactive aggregate in investigations of alkali-silica reaction. Results of these studies and recommendations for additional tests are presented in this report.

2. STANDARD REACTIVE MATERIALS

To serve as a standard reactive aggregate in tests such as ASTM C 441, certain criteria should be met. The material should

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[2] Certain trade names and company products are identified to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products are necessarily the best available for the purpose.

[3] In 1984 it appeared that Pyrex would not be available in the form of lump cullet as specified in ASTM C 441. Subsequently, however, the ASTM Subcommittee C09.02.02 was advised that the Pyrex would be available in this form.

[4] Formerly the National Bureau of Standards.

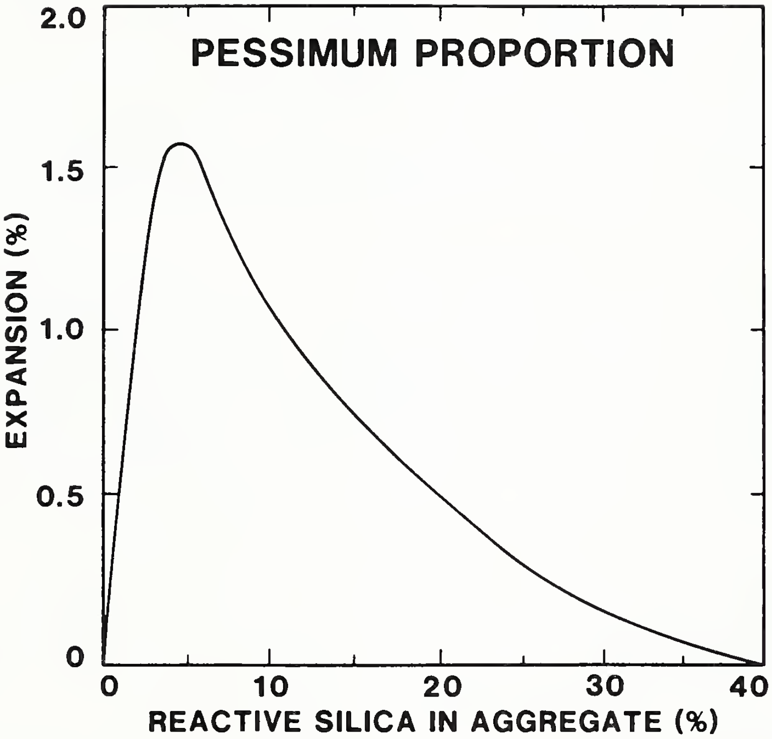

Figure 1. Schematic representation of pessimum proportion (Figure 6.8 of Mindess and Young [27]).

produce a high level of expansion in mortar bars, at least approximately 0.1 percent expansion when used with a cement containing approximately 1.0 percent total alkalies as equivalent sodium oxide (Na_2O). It should produce this expansion rapidly (i.e., within 14 days at 37.8°C) to allow accelerated testing. If the expansion shows a pessimum relationship to the proportion of aggregate that is reactive, the peak in expansion must be reasonably broad (here termed a flat pessimum) so that expansion results are not sensitive to small changes in the ratio of alkali to reactive aggregate. Expansion must be reduced considerably when measured using low-alkali cement and pozzolanic materials, so that the objectives of ASTM C 441 are met. Finally, the material must be homogeneous within each batch and uniform from batch-to-batch to assure reproducible expansion data.

Pyrex glass has been used as a standard reactive material in measuring expansion due to ASR since 1947. It was specified as a reactive material for mortar-bar expansion tests in the Bureau of Reclamation Specification No. 1904 in 1947 covering the performance of calcined reactive siliceous material for the Davis Dam. Stanton in 1950 [5] discussed the use of Pyrex glass sand and presented expansion results at 70°F and at 100°F at ages up to 1 year for mortars containing cement and cement plus pozzolanic material. The first publication of ASTM C 441, which utilizes Pyrex glass as the standard reactive material, was in 1959.

Notwithstanding its long history, Pyrex is not satisfactory as a standard reactive material in ASR studies. Stark [6] reported that two samples of Pyrex glass produced variable flow levels in an osmotic cell, which measures extent of deleterious reaction, and variable mortar bar expansion results. Barneyback [7] noted that Pyrex may produce variable expansion levels. Thus there have been cases where the expansion produced by Pyrex is not reproducible. The sodium content of Pyrex, approximately 4 percent Na_2O [8], probably also makes it unsuitable as a standard reactive material. In a recent study of ASR mechanisms, Struble [1] concluded that expansion appears to be a function of the extent of dissolution of aggregate in the cement pore solution, which depends on solution alkalinity, and is further influenced by the proportions of alkalies in solution, affecting composition and swelling behavior of the reaction product. Based on this understanding, one would expect considerable differences in expansion behavior between a material releasing appreciable alkali as it dissolves, such as the Pyrex glass, and a material releasing little or no alkali, such as most naturally occurring reactive aggregates. Figg [9] similarly concluded that Pyrex is not appropriate because it will provide additional alkali as it dissolves. He reported that glasses containing alkali and silica in appropriate proportions can produce expansion even with low-alkali cements.

The expansion behavior of Pyrex has been shown to differ in a number of ways from the behavior of naturally occurring aggregates. Brandt [10] noted that the behavior of Pyrex is different from the behavior of other reactive aggregates in that the glass is much more rapidly reactive, which means its expansion occurs before all the so-called active alkalies had been released from cement. Nixon and Gaze [11] reported that expansion produced by Beltane opal shows the typical pessimum relationship with the proportion of reactive material, whereas with Pyrex there was no pessimum, but rather a progressive increase in expansion as more Pyrex was used. In subsequent studies, the same authors [12] discussed differences in behavior between Pyrex and opal with respect to the reduction in expansion in mortars containing fly ash. With Pyrex, this reduction in expansion correlated with pozzolanic activity of fly ash, and the alkali content of ash had only a secondary effect. With opal, on the other hand, the alkali content of the fly ash had a substantial influence on reduction in expansion.

A few other materials have been used as standard reactive materials in ASR studies. One is Beltane opal, proposed by Diamond and Barneyback in 1976 [13]. It is a hydrothermally altered rhyolite collected by Barneyback from an abandoned open-pit mine in California [7]. However, Figg [9] noted that neither Pyrex nor Beltane opal model behavior of real, reactive aggregates in concrete. In particular, Beltane opal shows a particularly sharp pessimum relationship; i.e., high levels of expansion occur only over a very narrow range of opal content. For example, in one case the pessimum proportion found in our previous studies was approximately 2 percent of the total aggregate, and expansion was reduced to roughly 25 percent of its maximum value when the proportion of opal was decreased to 1 percent or increased to 4 percent [1,14].

Fused silica has also been proposed as a standard reactive material by both Figg [9] and Swamy and Al-Asali [15]. The latter reported that fine (50 percent passing 0.6 mm sieve) fused silica produced the most expansion when present at approximately 15 percent of the total aggregate.

More recently, calcined flint was proposed as a standard reactive aggregate by Lumley and Kollek [16]. They reported that the material is approximately 96 percent cristobalite, that its pessimum level is roughly 10 percent of the total aggregate, and its expansion level and rate are suitable for a standard reactive aggregate.

3. EXPERIMENTAL PROCEDURE

3.1 Materials

Two Type I cements, high- and low-alkali[5], were utilized for these studies. The high-alkali cement is Portland Cement Sample No. 72 from the Proficiency Sample Program of the Cement and Concrete Reference Laboratory at NIST. The low-alkali cement was obtained from the manufacturer, and was used in a previous study[6]. Alkali levels are listed in Table 1.

A number of reactive materials were studied, both manufactured glasses and naturally occurring materials. Each was used in combination with limestone for mortar-bar expansion tests. The proportion of limestone replaced by each reactive material was varied so as to determine at least approximately the pessimum level of each reactive material.

The limestone[7] was used in previous ASR studies [1,14]. As discussed [1], the limestone was selected from a quarry with no known history of alkali reaction. It was thought to contain only low levels of dolomite, which is important because high levels of dolomite and clay phases may indicate susceptibility to alkali-carbonate reaction. Subsequent analyses showed that some samples did contain appreciable dolomite, and possible effects of the dolomite are discussed later in this report.

Pyrex glass[8] was obtained as a lump cullet[9]. Pyrex is a sodium borosilicate glass, and typically contains 81.0 percent SiO_2, 13 percent B_2O_3, 4 percent Na_2O, and 2 percent Al_2O_3 [8].

Porous silica glass[10] was obtained in the form of rods, and is not available as a cullet. It is an intermediate material in

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[5] The designations Type I and high-alkali cement are described in ASTM C 150.

[6] This cement was designated as Cement G by Struble [1].

[7] Obtained from Martin Marietta Aggregates Company, Pinesburg, MD.

[8] No. 7740, obtained from Corning Glass Works, Corning, NY.

[9] Lump cullet is a term that is applied to waste glass from the manufacturing process, either to the residual melt left during a batch process or to broken pieces of finished glass, typically rods and tubes, that are added to the melt.

[10] No. 7930, obtained from Corning Glas Works, Corning, NY.

Table 1. Cement alkali levels

Cement	Na_2O (% by mass)	K_2O (% by mass)
High-alkali[a]	0.28	1.02
Low-alkali[b]	0.20	0.31

[a] Reported by CCRL [26].
[b] Reported by Struble [1].

the manufacture of Vycor glass, which begins with removal of the non-silica constituents of a borosilicate glass by a chemical leaching process, producing the porous silica glass. Porous silica glass is expected to have a relatively high solubility rate due to its high specific surface area (approximately 150 m^2/g) and small (approximately 5.0 nm) pore diameter [17]. The Vycor glass[11] was obtained as broken tubing with a typical wall thickness of 1 mm, and is not available as lump cullet. Both glasses contain approximately 96 percent SiO_2, and the remaining 4 percent is largely B_2O_3 [18].

The fused quartz[12] glass was also in the form of broken tubing. This glass is produced by melting material from a high-purity quartz deposit [19].

The fused silica[13] was obtained as industrial-grade glass, though it is also available as cullet. The fused silica is a high-purity, high-quality optical glass, produced by flame hydrolysis of $SiCl_4$ [20].

The calcined flint[14] was obtained as particles 1 mm to 2 mm in size. It is made by acid-washing a commercial flint, then firing it at 1375°C for 6 hours [16].

The Beltane opal[15] was used in other studies in our laboratory [1,14]. Gutteridge and Hobbs [21] reported that the reactive components of Beltane opal are an amorphous, opaline constituent and α-cristobalite.

The novaculite[16] was also used in other studies in our laboratory [1,14]. It is a fine-grained orthoquartzite, composed largely of SiO_2. This material produced only slight expansion

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[11] No. 7913, obtained from Corning Glass Works, Corning, NY.

[12] Obtained from General Electric, Quartz Department, Cleveland, OH.

[13] Obtained from Corning Glass Works, Corning, NY.

[14] Obtained from Blue Circle Industries PLC, Technical Services Division, Greenhithe, UK.

[15] Obtained from Professor S. Diamond, Purdue University, W. Lafayette, IN.

[16] Obtained from Ward's Natural Science Establishment Inc., Rochester, NY.

when tested at room temperature, and was included in the present studies in case it produced more substantial expansion at 37.8°C.

The rhyolite[17] sand had been used as a reactive material in another laboratory [22]. The sand is crushed rhyolite from a quarry in Arizona and is used in light-weight concrete and masonry [23].

3.2 <u>Mortar Bar Expansion Tests</u>

The aggregate materials were crushed as necessary, sieved, and reconstituted to obtain the grading specified in ASTM C 441 (Table 2). In mixes containing reactive material, limestone was replaced by the desired proportion (by mass) of the reactive constituent. Except for the calcined flint (which had too fine a particle size distribution), the reactive material replaced limestone in each size fraction; for the calcined flint, no replacement could be made in the two coarser fractions, so a correspondingly larger amount of its coarsest fraction (1.18 mm to 0.60 mm) was used.

In general, the mortar bars were prepared according to ASTM C 441 and ASTM C 227-87, Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method). The following amounts were used for each batch: 300 g cement, 675 g sand, and 145.5 mL water, for a water-to-cement ratio (w/c) of 0.485. (These amounts are slightly lower than the amounts specified in ASTM C 441, but the ratio of sand-to-cement is the same.) A constant water content was used in the present study, whereas a variable water content is specified in ASTM C 441 to produce flow within a specified range.

The mortars prepared for these studies are described in Table 3. The proportions of reactive aggregate and non-reactive limestone were varied to extend above and below the pessimum proportion for each reactive material. One batch of each mortar was prepared to provide two specimens of each mixture.

Bars were removed from the mold after 24 hours, then stored throughout the test period in containers and racks described in C 227. The sealed container allows vertical storage of the bars in a moist environment. As specified in ASTM C 227 and C 441, bars were stored at 37.8°C throughout the expansion test. Length changes were measured periodically at room temperature using a length comparator.

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[17] Obtained from Johnson-Stewart-Johnson Mining Co., Mesa, AZ.

Table 2. Aggregate grading[a]

Sieve Size Passing	 Retained on	Amount (% by mass)
No. 4 (4.75 mm)	No. 8 (2.36 mm)	10
No. 8 (2.36 mm)	No. 16 (1.18 mm)	25
No. 16 (1.18 mm)	No. 30 (0.60 mm)	25
No. 30 (0.60 mm)	No. 50 (0.30 mm)	25
No. 50 (0.30 mm)	No. 100 (0.15 mm)	15

[a] According to ASTM C441, Table 1.

Table 3. Expansion results

ID	Reactive Material	Proportion of aggregate (% by mass)	Cement[a]	Final Expansion (% by length)
A2	none	0	H	0.04
A3-2	none	0	H	0.00
B1	Pyrex	50	H	0.46
B3	Pyrex	50	L	0.02
A3	Pyrex	80	H	0.46
A1	Pyrex	100	H	0.68
F5	porous silica	2	H	0.03
F2	porous silica	10	H	0.01
F1	porous silica[b]	100	H	0.14
F3	Vycor	10	H	0.30
F4	Vycor	50	H	0.88
F6	Vycor	75	H	0.75
F7	Vycor-2[c]	100	H	0.98
F9	Vycor-2[c]	100	L	0.32
L2	fused quartz	50	H	0.85
L3	fused quartz	50	L	0.31
L1	fused quartz	100	H	0.99
K2	fused silica	50	H	0.67
K3	fused silica	50	L	0.27
K1	fused silica	100	H	0.81
M5	calcined flint	10	H	0.14
M2	calcined flint	10	L	0.01
M4	calcined flint	15	H	0.25
M1	calcined flint	15	L	0.01
M6	calcined flint	20	H	0.27
M3	calcined flint	20	L	0.01
M7	calcined flint	30	H	0.32
M8	calcined flint	50	H	0.25
D2	opal	1	H	0.09
D1	opal	2	H	0.28
E1	opal	4	H	0.24
D3	opal	6	H	0.20
G1	novaculite	10	H	0.03
G2	novaculite	20	H	0.03
G3	novaculite	50	H	0.00
C3	rhyolite	10	H	0.00
B2	rhyolite	50	H	0.03
C2	rhyolite	100	H	0.02

[a] Cements designated as H, high-alkali, or L, low-alkali.
[b] Extra water (150 ml) was added to this mortar during mixing.
[c] Second batch of glass.

4. RESULTS

Expansion curves for mortars using the high-alkali cement are shown in Figs. 2 through 10. Expansion generally began immediately and increased most rapidly during the initial period. After a few weeks, the length gradually leveled off. The expansion levels at 2 months were usually as high or nearly as high as the final level (~6 months to 2 years). The only material that was moderately slow to reach its final expansion was the porous silica glass (Fig. 3).

The final expansion levels (Table 3) with high-alkali cement vary considerably for the various reactive materials studied. The naturally occurring materials (novaculite and rhyolite) produced low expansion, generally less than 0.05 percent. Calcined flint, Beltane opal and the porous silica glass produced moderate expansion levels, 0.10 percent to 0.30 percent. The other commercial glasses (Vycor, fused quartz, and fused silica) produced high expansion levels, nearly 1.0 percent, greater than the approximately 0.7 percent expansion produced by Pyrex.

Although these studies did not include enough replacement levels to determine precisely the pessimum proportion for each material, the results provide an indication of the pessimum level. All the manufactured glasses appear to have a high pessimum proportion, in the range 50 percent to 100 percent, and a flat pessimum, such that appreciable variation in proportion of reactive material (e.g., 10 percent) caused little variation in expansion. This pattern was followed by Pyrex (Fig. 2), porous silica (Fig. 3), and Vycor (Fig. 4), and probably by fused quartz (Fig. 5) and fused silica (Fig. 6); the latter two were tested only at 50 and 100 percent but appear similar. Beltane opal (Fig. 8), on the other hand, shows a very low pessimum at approximately 2 percent, as was reported previously [1,14]. The Beltane opal also shows an extremely sharp pessimum relationship, such that even small variations in the proportion of reactive material (e.g., 4 percent) causes much variation in expansion. The calcined flint (Fig. 7) shows an intermediate pessimum level, approximately 30 percent and a flat pessimum. Expansion levels for the novaculite and rhyolite were very low, less than the limestone, thus probably not due to alkali-silica reaction and not showing a pessimum effect.

There was considerable difference among the reactive materials in the degree to which expansion was reduced using a low-alkali cement (Figs. 11-15). With Pyrex (Fig. 11), the expansion level using the low-alkali cement was only 4 percent of the level using the high-alkali cement. With Vycor, fused quartz, and fused silica (Figs. 12-14), the effect was not so great; the expansion levels using the low-alkali cement were approximately 30 percent to 40 percent of the expansion levels using the high-alkali cement. The behavior with calcined flint

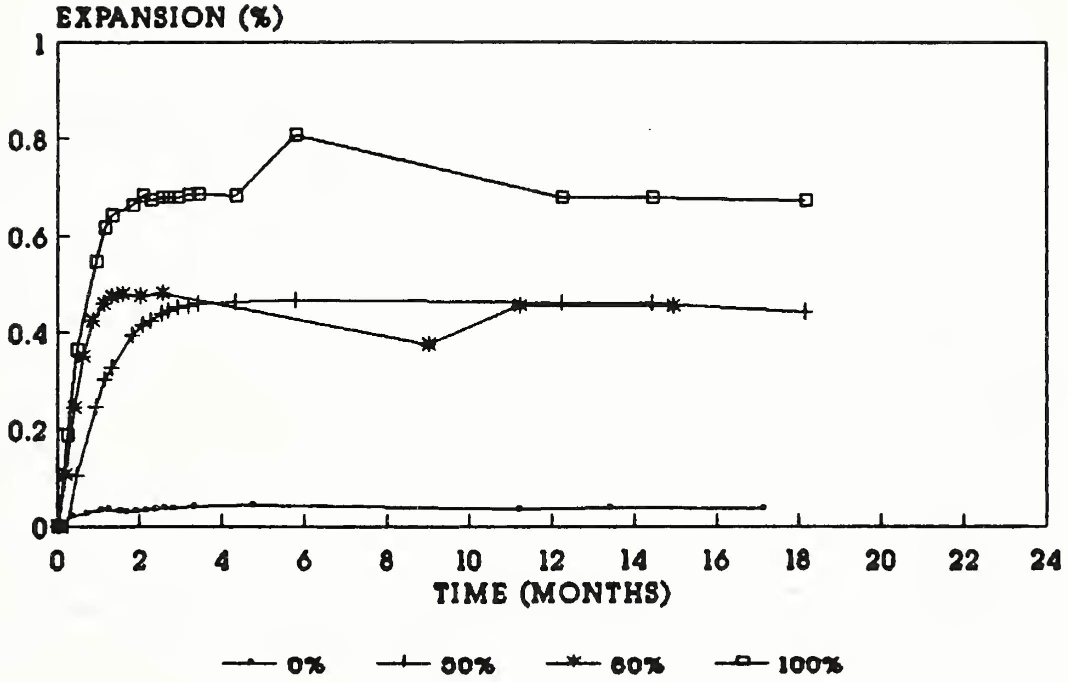

Figure 2. Mortar-bar expansion using Pyrex glass and high-alkali cement, with various proportions of glass replacing limestone.

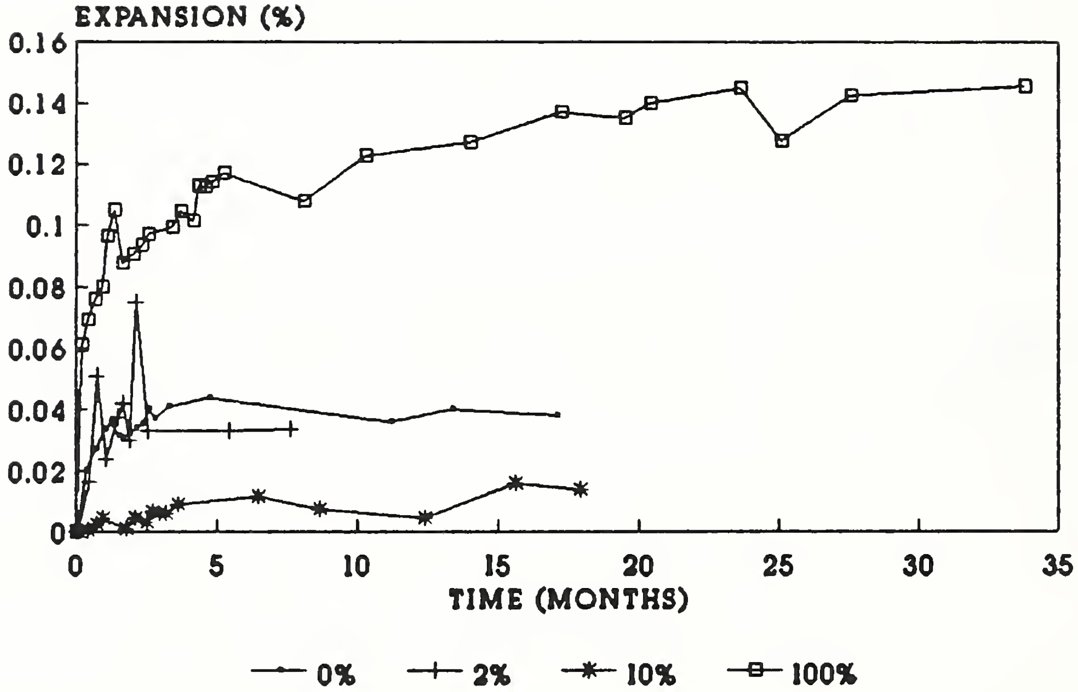

Figure 3. Mortar-bar expansion using porous silica glass and high-alkali cement, with various proportions of glass replacing limestone.

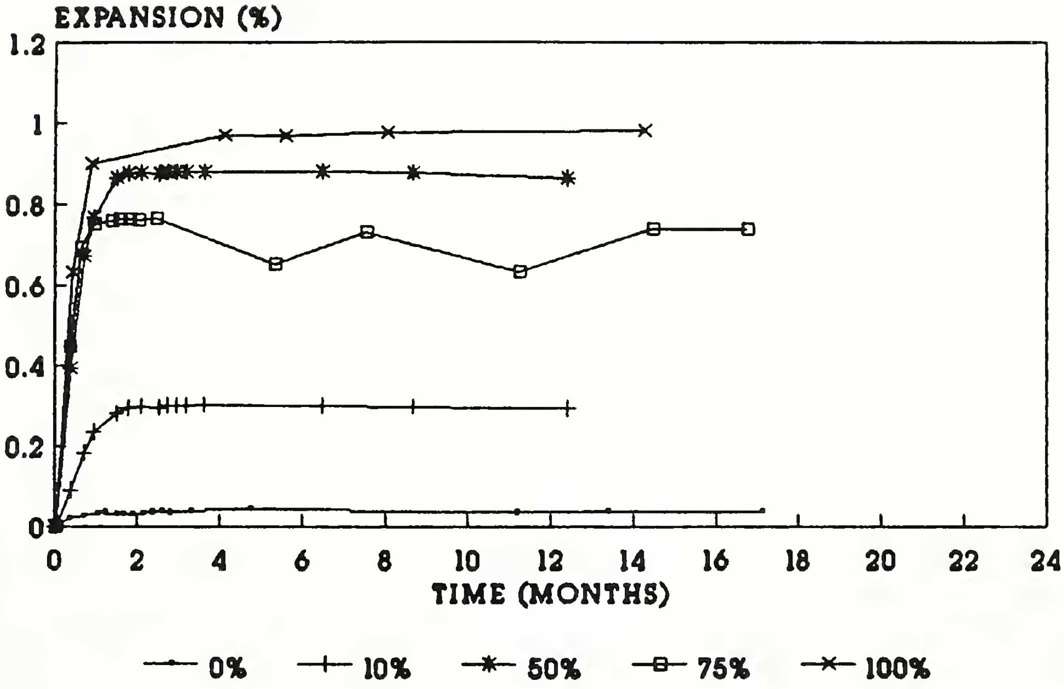

Figure 4. Mortar-bar expansion using Vycor glass and high-alkali cement, with various proportions of glass replacing limestone.

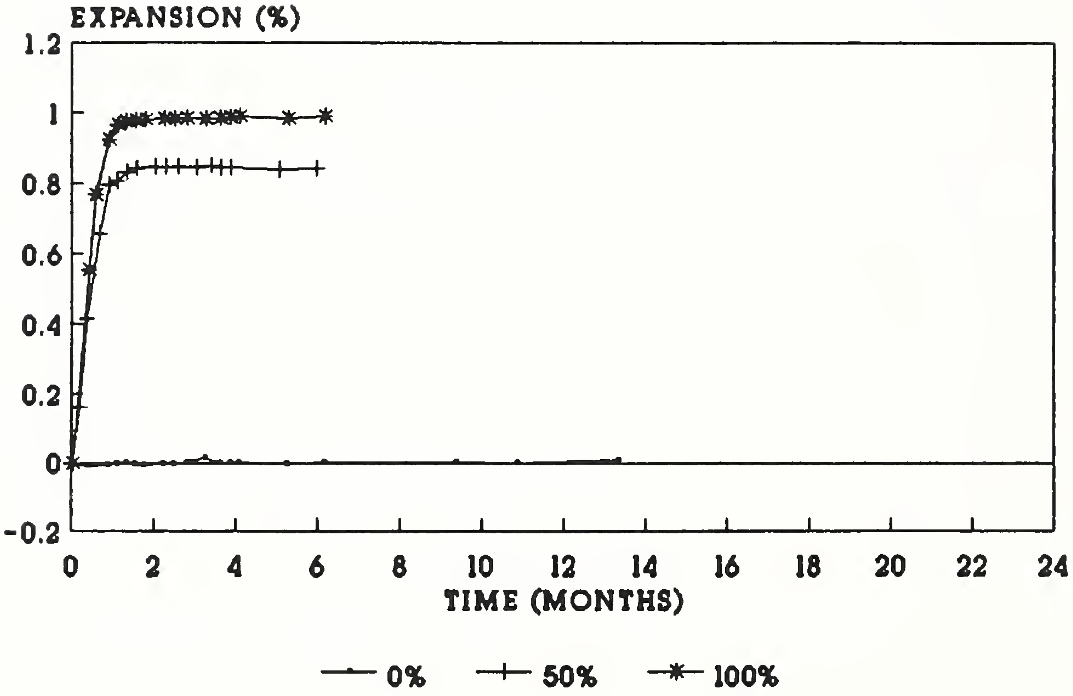

Figure 5. Mortar-bar expansion using fused quartz glass and high-alkali cement, with various proportions of glass replacing limestone.

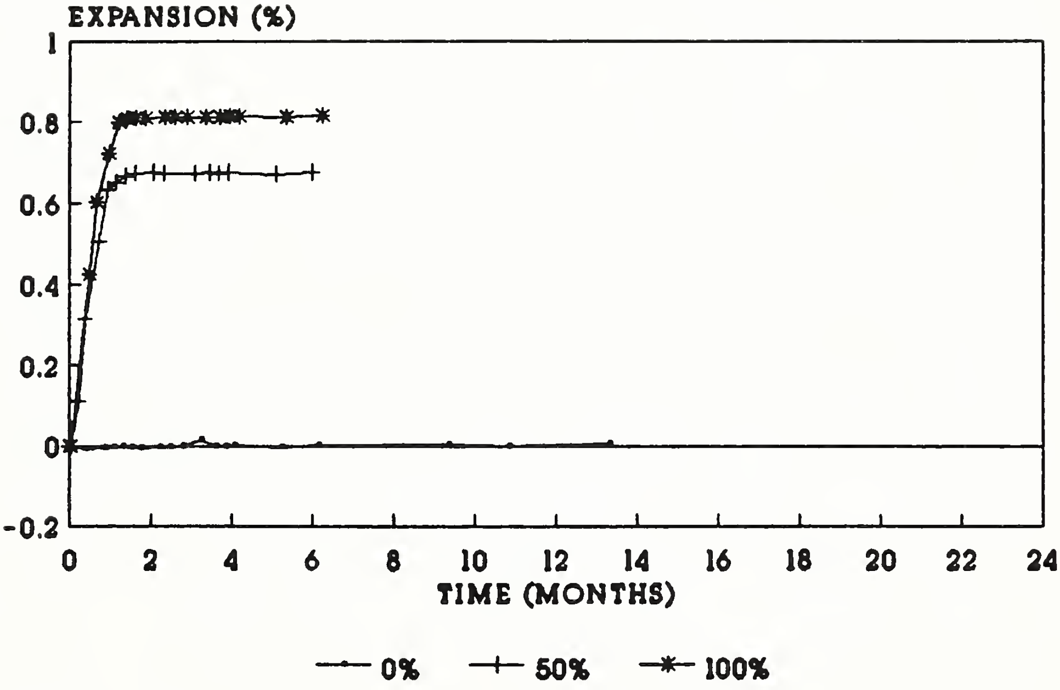

Figure 6. Mortar-bar expansion using fused silica glass and high-alkali cement, with various proportions of glass replacing limestone.

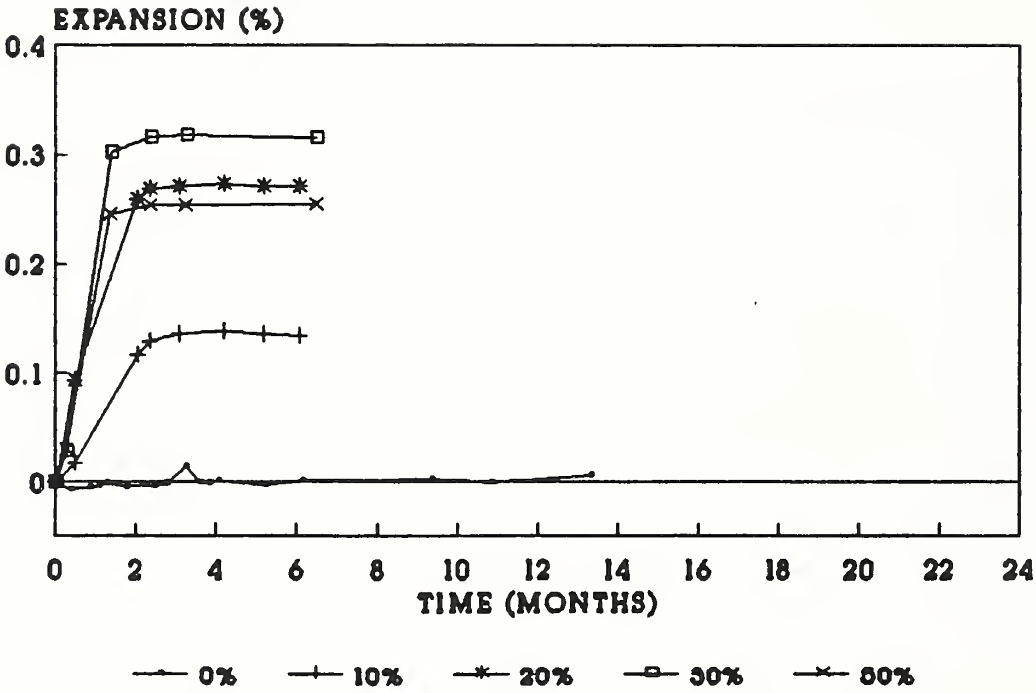

Figure 7. Mortar-bar expansion using calcined flint and high-alkali cement, with various proportions of flint replacing limestone.

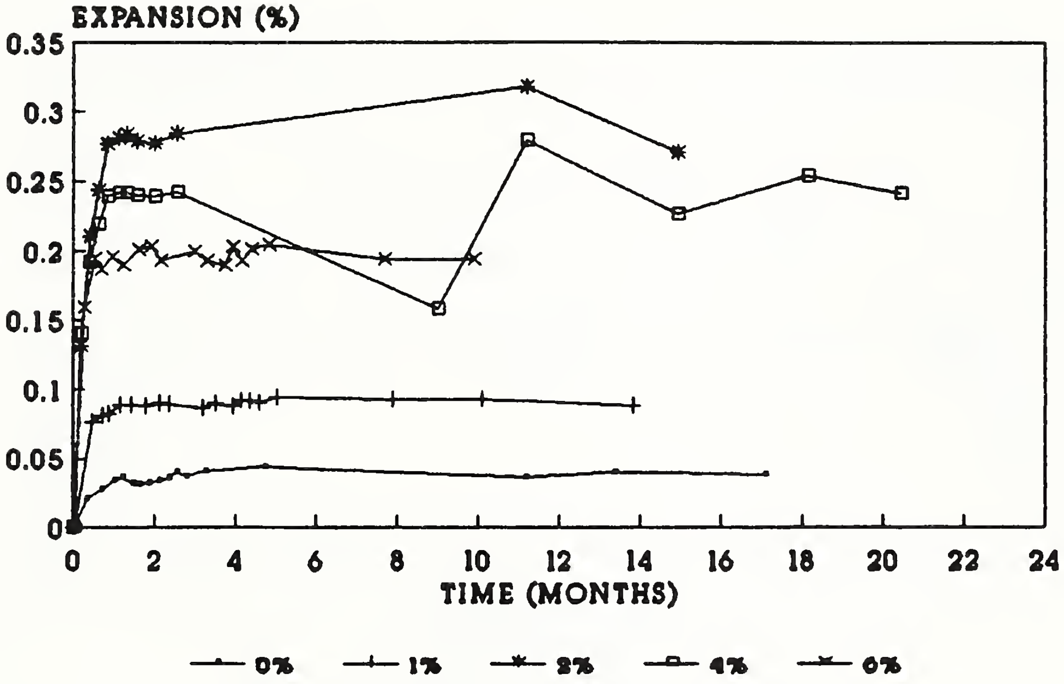

Figure 8. Mortar-bar expansion using Beltane opal and high-alkali cement, with various proportions of opal replacing limestone.

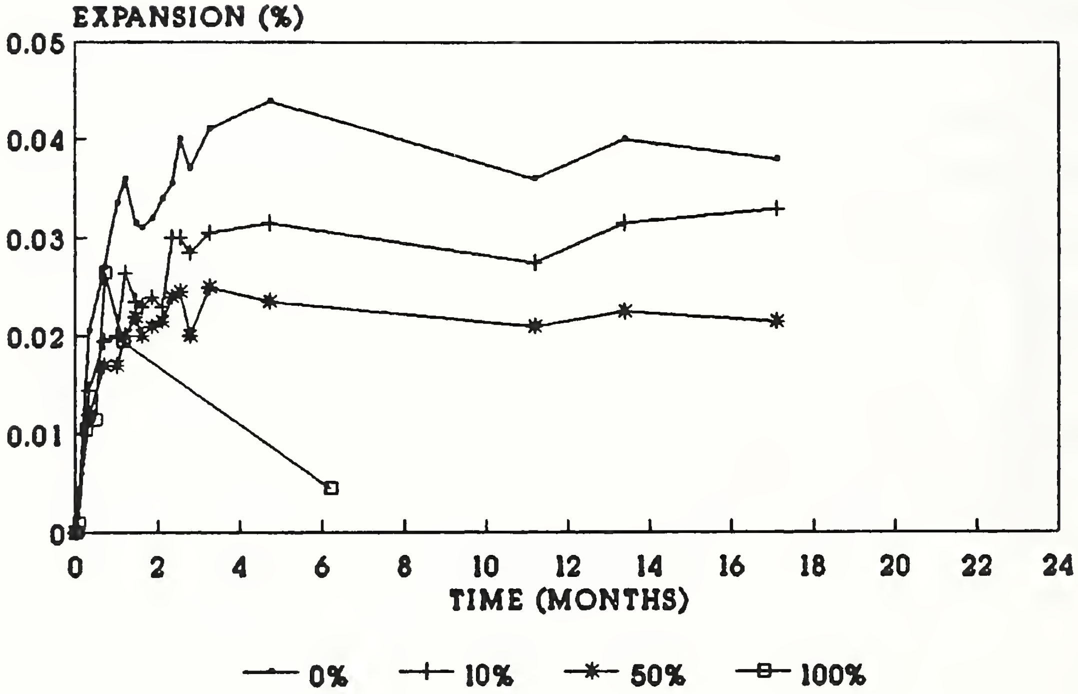

Figure 9. Mortar-bar expansion using novaculite (orthoquartzite) and high-alkali cement, with various proportions of quartzite replacing limestone.

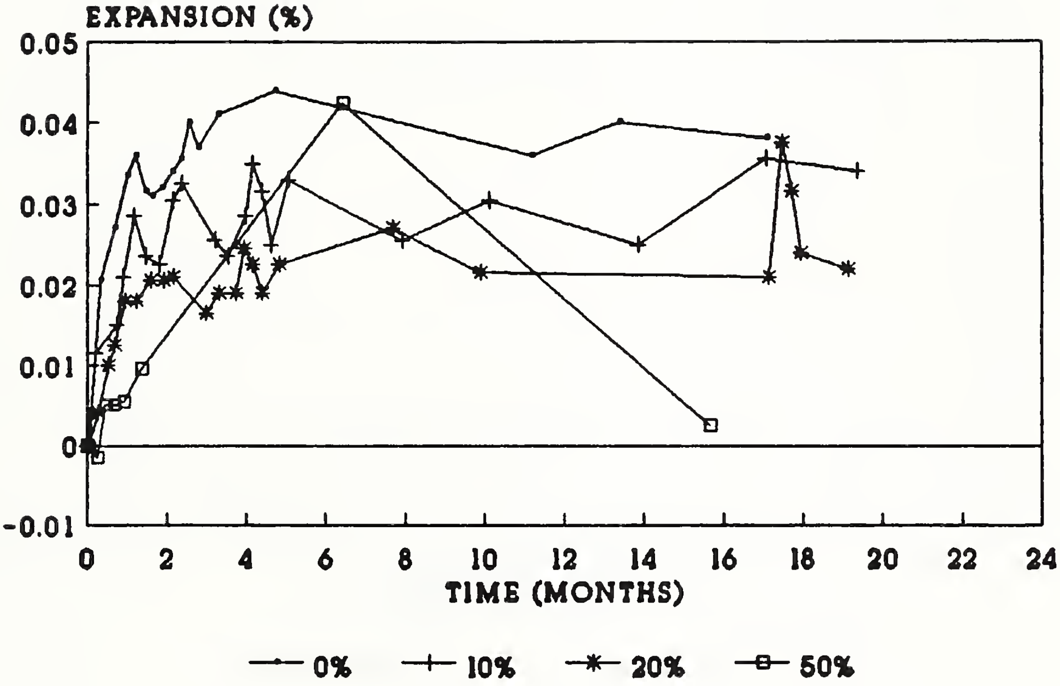

Figure 10. Mortar-bar expansion using rhyolite and high-alkali cement, with various proportions of rhyolite replacing limestone.

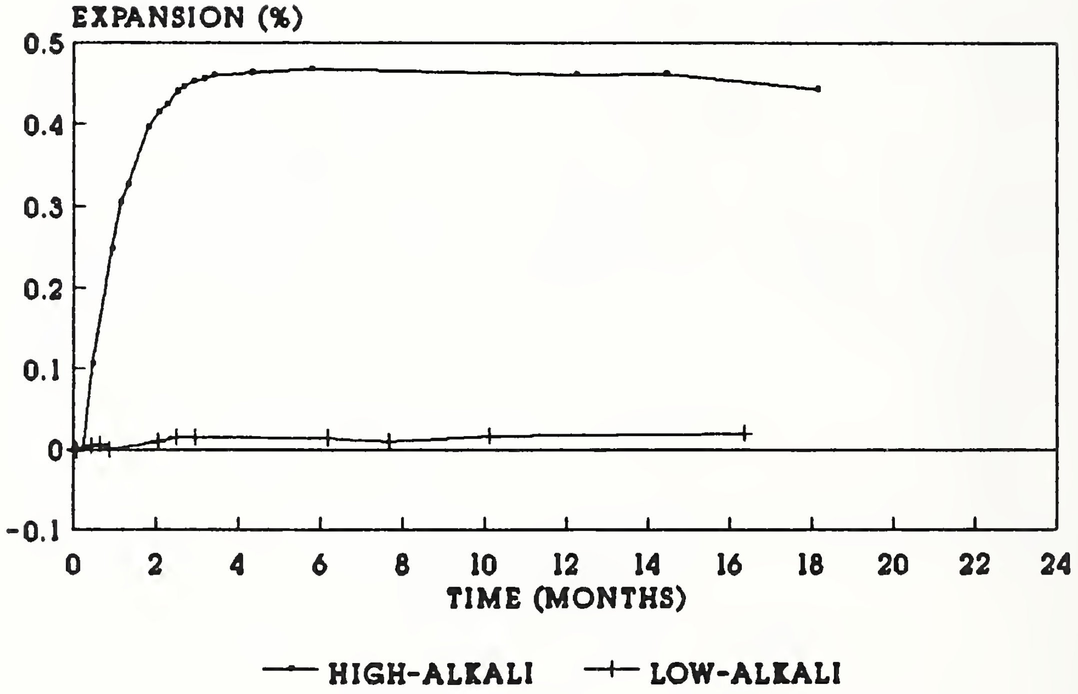

Figure 11. Mortar-bar expansion using Pyrex glass and high- or low-alkali cement (limestone plus 50 percent glass).

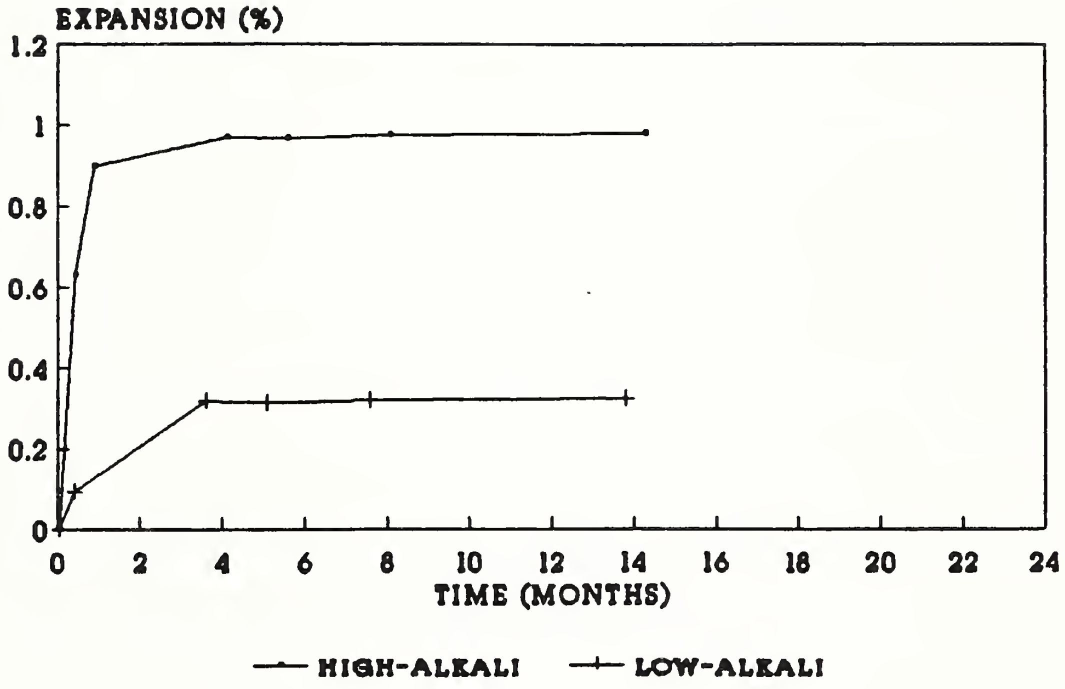

Figure 12. Mortar-bar expansion using Vycor glass and high- or low-alkali cement (100 percent glass).

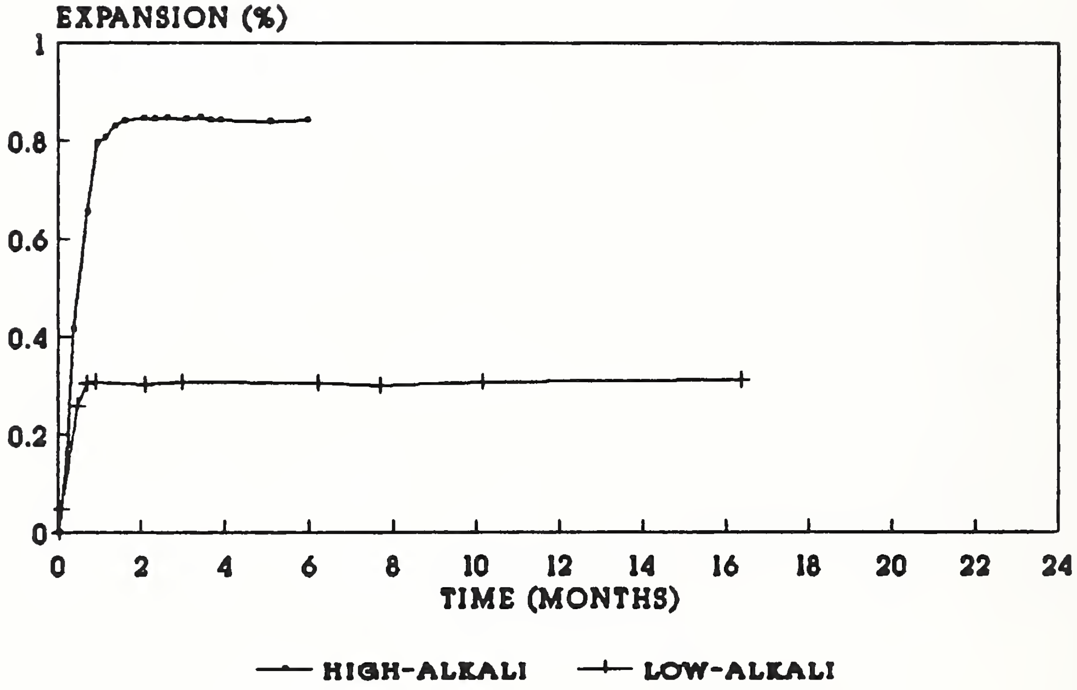

Figure 13. Mortar-bar expansion using fused quartz glass and high- or low-alkali cement (limestone plus 50 percent glass).

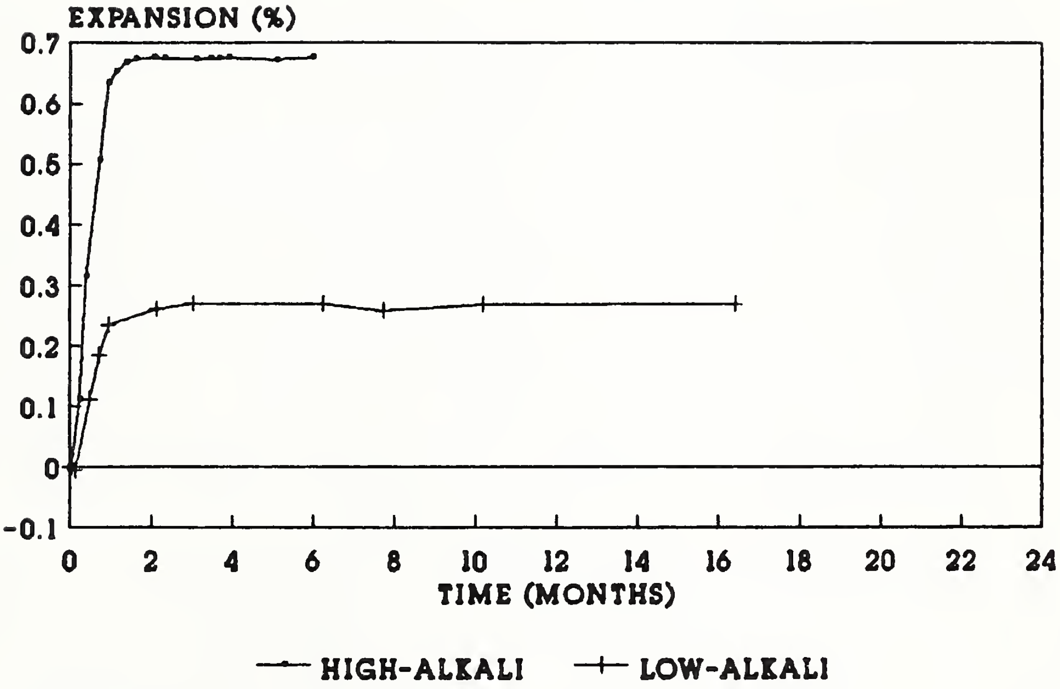

Figure 14. Mortar-bar expansion using fused silica glass and high- or low-alkali cement (limestone plus 50 percent glass).

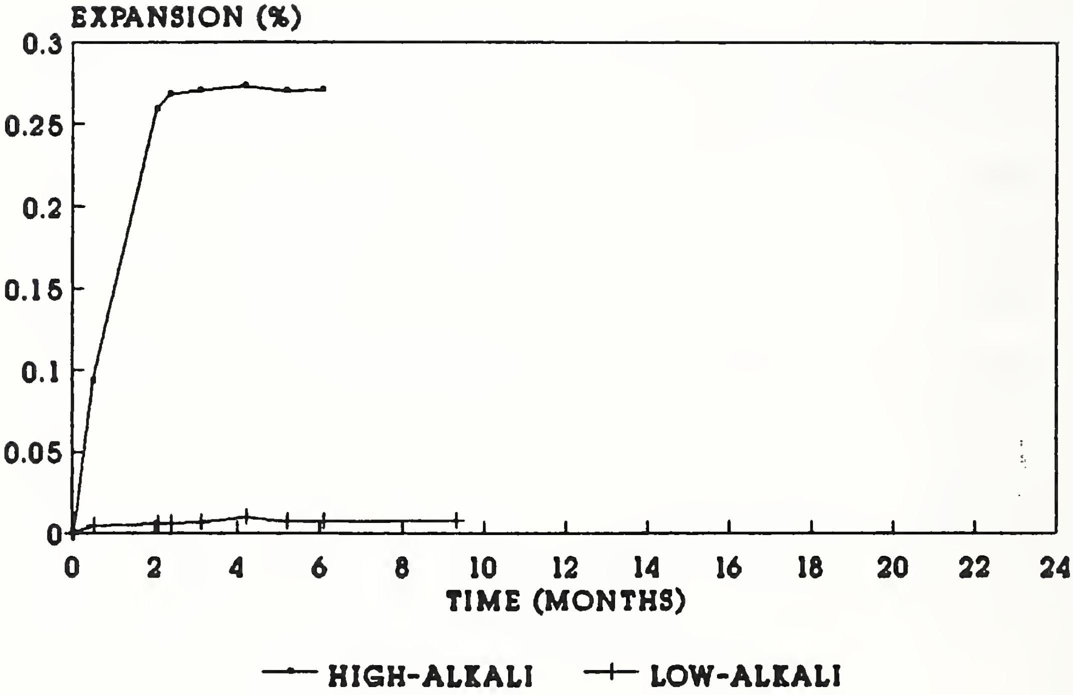

Figure 15. Mortar-bar expansion using calcined flint and high- or low-alkali cement (limestone plus 20 percent flint).

(Fig. 15) was similar to Pyrex; the expansion level using the low-alkali cement was extremely low relative to the level using the high-alkali cement.

5. DISCUSSION

As discussed previously, it was found after some experiments had been carried out that one batch of limestone contained appreciable dolomite. Regardless of which limestone was used, mortars containing 100 percent limestone showed very little expansion. Sample A2 (Table 3) used limestone containing dolomite; its final expansion level was 0.04 percent. Sample A3-2 (Table 3) used a later batch of limestone containing no dolomite; its final expansion level was 0.00 percent. The latter level is significant, but still quite low. Therefore, it is assumed that the dolomite did not affect the expansion results to any great extent. The plots of expansion data for each aggregate (Figs. 2-10) utilized whichever of the two batches was used to prepare the mortars containing reactive material.

In assessing the suitability of each candidate reactive material, we consider the expansion level and rate, the sensitivity of expansion to proportion of aggregate, the extent to which expansion is reduced using a low-alkali cement, and the expected reproducibility. As discussed earlier, Pyrex is not a good candidate for two principal reasons, its lack of reproducibility and its high sodium content. Five materials produced a high final expansion level and a high initial expansion rate of expansion: Vycor, fused quartz, fused silica, opal, and calcined flint. Of these, the opal is rejected as a candidate because its pessimum proportion is so low and sharp. A sharp pessimum is undesirable because the material would have to be tested at many replacement levels to define fully the relationship between expansion and proportion of reactive material for a specific cement.

The remaining candidate standard reactive materials varied somewhat in the extent to which their expansion was influenced by the alkali content of the cement. As is desirable for a standard reactive material, Pyrex produced high expansion with high-alkali cement and very low expansion with low-alkali cement. This was not the expected behavior. Following the reasoning of Figg [9], we expected that the sodium ions provided by Pyrex would make it relatively insensitive to the alkali level in the cement. The calcined flint also produced very low expansion with low-alkali cement. On the other hand, the other three commercial glasses, Vycor, fused quartz, and fused silica, produced intermediate expansion with low-alkali cement. Thus, these three commercial glasses appear somewhat less suitable than Pyrex and calcined flint as standard reactive materials.

Although we have not assessed the batch-to-batch reproducibility of the candidate materials, certain production parameters are expected to affect reproducibility. For example, Pyrex lump cullet is sometimes annealed and sometimes quenched with water [24], either of which may affect reactivity. Such variability in processing may be responsible for the poor batch-to-batch reproducibility discussed earlier. A lump cullet, with variable thermal history, is therefore not a good choice for a standard reactive aggregate. Vycor cannot be tempered [25], fused quartz is not annealed during its manufacture [19], and fused silica is likewise not annealed [20]. Therefore these three materials are expected to have better batch-to-batch reproducibility. A second batch of Vycor was included in this study, however, and produced somewhat different expansion levels than the first batch. Finally, though calcined flint is derived from a naturally occurring material, and the homogeneity of a naturally occurring material is difficult to assure, the processing involved in the manufacture of calcined flint is expected to remove constituents that would otherwise cause heterogeneity.

6. ADDITIONAL STUDIES

Before a material can be proposed as a standard, the following additional studies are recommended. Mortars need to be tested using high- and low-alkali cement and fly ash. The pessimum proportion should be determined for each cement to assure that reductions in expansion with low-alkali cement or fly ash are not indirect effects of changes due to pessimum proportion. Several batches of each material should be tested to determine the long-term (batch-to-batch) reproducibility. Finally, consideration must be given to the long-term availability, to possible availability in the size grading specified in ASTM C441, and to the cost.

7. CONCLUSIONS

Because of its reported poor reproducibility and its sodium content, Pyrex glass, which is used in ASTM C 441, is not satisfactory as a standard reactive material in alkali-silica investigations. Preliminary studies have been carried out to identify materials to use in place of Pyrex. Results of mortar-bar expansion tests were as follows. With high-alkali cement, calcined flint produced moderate expansion levels and a rapid early rate of expansion, and with low-alkali cement it produced very little expansion. With high-alkali cement, Vycor, fused quartz, and fused silica produced high expansion levels and a rapid early rate of expansion, but with low-alkali cement they produced moderate expansion. Based on these results, it is concluded that calcined flint, Vycor, fused quartz, and fused silica have good potential, and that calcined flint offers the best potential as a standard reactive material in alkali-silica reaction investigations. Additional investigations are needed to assess reproducibility and to demonstrate that expansion is reduced by using low-alkali cements or cement plus mineral admixture.

8. ACKNOWLEDGEMENTS

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9. REFERENCES

1. L. Struble, The influence of cement pore solution on alkali-silica reaction. PhD thesis, Purdue University. NBSIR 87-3632, 265 pages. Washington: National Bureau of Standards (US) (1987).

2. S. Diamond, A review of alkali-silica reaction and expansion mechanisms 1. Alkalies in cements and in concrete pore solutions. Cement and Concrete Research, 5(4), 329-346 (1975).

3. S. Diamond, A review of alkali-silica reaction and expansion mechanisms 2. Reactive aggregates. Cement and Concrete Research, 6(4), 549-560 (1976).

4. S. Diamond, Chemical Reactions other than Carbonate Reactions. In: Significance of Tests and Properties of Concrete and Concrete-Making Materials, ASTM STP 169B, Chapter 40, pp. 708-721. Philadelphia: ASTM (1978).

5. T.E. Stanton, Studies of use of pozzolans for counteracting excessive concrete expansion resulting from reaction between aggregates and the alkalies in cement. In: Symposium on Use of Pozzolanic Materials in Mortars and Concretes, STP 99, pp. 178-201. Philadelphia: ASTM (1950).

6. D. Stark, Osmotic cell test to identify potential for alkali-aggregate reactivity. In: Alkalis in Concrete, edited by G.M. Idorn and S. Rostam, Proceedings of the Sixth International Conference, held at the Technical University of Denmark, June 22-25, 1983, pp. 351-357. Copenhagen: Danish Concrete Association (1983).

7. R.S. Barneyback, Jr., Alkali-silica reaction in portland cement concrete. PhD thesis, Purdue University, 352 pages (1983).

8. Personal communication from Mr. A. Flood, Corning Glass Works, Corning NY, to Leslie Struble, National Bureau of Standards, Gaithersburg MD, April 9, 1984.

9. J.W. Figg, Reaction between cement and artificial glass in concrete. In: Proceedings of the Fifth International Conference on Alkali-Aggregate Reaction in Concrete, edited by R.E. Oberholster, paper S252/7, pages 1-18. Pretoria: National Building Research Institute of the Council for Scientific and Industrial Research (1981).

10. M.P. Brandt, discussion to paper S252/10. In: Alkali-Aggregate Reaction, edited by R.E. Oberholster, Proceedings of the Fifth International Conference on Alkali-Aggregate Reaction in Concrete, p. 7. Pretoria: National Building Research Institute of the Council for Scientific and Industrial Research (1981).

11. P.J. Nixon and M.E. Gaze, The use of flyash and granulated blastfurnace slag to reduce expansion due to alkali-aggregate reaction. In: Alkali-Aggregate Reaction, edited by R.E. Oberholster, Proceedings of the Fifth International Conference on Alkali-Aggregate Reaction in Concrete, paper S252/32, pages 1-10. Pretoria: National Building Research Institute of the Council for Scientific and Industrial Research (1981).

12. P.J. Nixon and M.E. Gaze, The effectiveness of flyashes and granulated blastfurnace slags in preventing AAR. In: Alkalis in Concrete, edited by G.M. Idorn and S. Rostam, Proceedings of the Sixth International Conference, held at the Technical University of Denmark, June 22-25, 1983, pp. 61-68. Copenhagen: Danish Concrete Association (1983).

13. S. Diamond and R.S. Barneyback, Jr., "Standard" alkali-reactive silica available. Cement and Concrete Research, 6(5), 726 (1976).

14. L. Struble and S. Diamond, Influence of cement alkali distribution on expansion due to alkali-silica reaction. In: Alkalies in Concrete, edited by V.H. Dodson, STP 930, pp. 31-45. Philadelphia: ASTM (1986).

15. R.N. Swamy and M.M. Al-Asali, Influence of alkali-silica reaction on the engineering properties of concrete. In: Alkalies in Concrete, edited by V.H. Dodson, STP 930, pp. 69-86. Philadelphia: ASTM (1986).

16. Lumley, J.S., and Kollek, J.J. 1987. Cristobalite as a reference reactive aggregate. Report No. PTN 87/82, Blue Circle Industries PLC, Technical Services Division, Greenhithe UK.

17. Personal communication from Mr. Paul Dohn, Corning Glass Works, Corning NY, to Leslie Struble, National Bureau of Standards, Gaithersburg MD, April 2, 1985.

18. Personal communication from Mr. A. Flood, Corning Glass Works, Corning NY, to Leslie Struble, National Bureau of Standards, Gaithersburg MD, June 29, 1984.

19. Personal communication from Mr. Vern Gogunke, General Electric Company, Cleveland OH, to Leslie Struble, National Bureau of Standards, April 10, 1985.

20. Personal communication from Mr. Herb Miska, Corning Glass Works, Corning NY, to Leslie Struble, National Bureau of Standards, Gaithersburg MD, April 4, 1985.

21. Gutteridge, W.A., and Hobbs, D.W. 1980. Some chemical and physical properties of Beltane opal rock and its gelatinous alkali silica reaction product. Cement and Concrete Research, 10(2), 183-193.

22. Personal communication from Mr. Fred Kinney, Master Builders, Cleveland OH, to Leslie Struble, National Bureau of Standards, June 22, 1984.

23. Personal communication from Mr. D.W. Tobey, Johnson-Stewart-Johnson Mining Company, Mesa AZ, to Leslie Struble, National Bureau of Standards, July 5, 1984.

24. Personal communication from Mr. T. Elmer, Corning Glass Works, Corning NY, to Leslie Struble, National Bureau of Standards, June 29, 1984.

25. Personal communication from Mr. Dan Lapp, Corning Glass Works, Corning NY, to Leslie Struble, National Bureau of Standards, Gaithersburg MD, February 12, 1986.

26. Cement and Concrete Reference Laboratory, Reference Sample Program, Final Report, April 13, 1984.

27. S. Mindess and J.F. Young, Concrete. 671 pages. Englewood Cliffs: Prentice-Hall, Inc. (1981).

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Preliminary studies have been carried out to identify candidate materials for use as a standard reactive aggregate in alkali-silica investigations. The materials studied included several commercial glasses, an opal, a quartzite, a rhyolite and a calcined flint. Candidate materials were tested according to for their expansion in mortars prepared using either a high-alkali or a low-alkali cement, a nonreactive limestone sand, and some proportion of reactive material. Tests were carried out according to ASTM C 441-81, Standard Test Method for Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction, and ASTM C 227-87, Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method). The proportion of limestone replaced by each reactive material was varied so as to bracket the pessimum proportion (i.e., the proportion of reactive material producing the highest level of expansion). Mortar-bar expansion levels were measured throughout reaction periods of approximately 6 months to 1 year. Expansion results are presented and discussed. With high-alkali cement, the calcined flint produced moderate expansion levels and a rapid early rate of expansion, and with low-alkali cement it produced very little expansion. With high-alkali cement, the Vycor, fused quartz, and fused silica, produced high expansion levels and a rapid early rate of expansion, but with low-alkali cement they produced moderate expansion. Based on these studies, the Vycor, fused quartz, fused silica, and calcined flint appear suitable as standard reactive materials; the calcined flint appears especially promising.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

aggregate; alkali-silica reaction; calcined flint; cement; concrete; expansion; fused quartz; fused silica; mortar-bar expansion; Pyrex; standard reactive aggregate; Vycor

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