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Compositional Variations Associated with Carbonate Aggregate Reactions

JOHN LEMISH, ROBERT J. HARWOOD, CARL L. HILTROP, and MICHAEL A. WERNER, respectively, Professor, Department of Geology, Iowa State University; Graduate Student, Stanford University; Assistant Professor, Department of Chemistry, Colorado School of Mines; and Research Assistant, Department of Geology, Iowa State University

Changes in the composition of aggregates and cement pastes were studied by placing cubes of various aggregates in cement paste bars and reacting them in a water bath at 130 F for a 3-mo period. Chemical and X-ray analyses were made on samples taken at specified intervals across the aggregate into the cement paste. The data indicate that much of the silica in the shell zone is locally derived from breakdown of quartz. Silica can migrate out of the rock into the cement paste or farther back into the rock. Dedolomitization is a concurrent reaction.

Silica migration was postulated to represent an equilibrium reaction controlled by the relative concentration of available silica in adjacent cement and aggregate and the local pH environment.

The results indicate that carbonate aggregate reactions represent an attempt of aggregates to come into equilibrium with the high pH environment of concrete. Silica migration and dedolomitization occur concurrently. Released calcium could react to form calcium carbonate. Silica in the shell zones can be locally derived from the aggregate itself when a cement paste medium is used for the reaction. Cement pastes are not a source of silica but may provide calcium and high pH environment.

• WITH THE recognition of the reactivity of some carbonate rocks used as coarse aggregate in concrete (1, 2, 3) a considerable amount of research has been conducted on such rock and its reaction in Iowa (1, 2, 4, 5, 6, 7) and elsewhere (3, 8, 9). To date most of the effort has been concentrated in an attempt to understand the nature of the reaction. Such knowledge is necessary before a causal relationship can be definitely established between deleterious behavior and carbonate aggregate reactions in concrete.

The Iowa State Highway Commission was able to correlate the poor service record of certain highways to the carbonate aggregates obtained from some quarries, notably the LeGrand and Glory. Past work which has been concentrated on intensive study of the Glory aggregate indicated the rock was an argillaceous dolomite with relatively high insoluble residue which reacted in highway concrete to form "reaction shells." Such concrete was generally characterized by progressive cracking, spalling away from joints, general weakening, and a soft carbonated matrix. Bisque (2) showed these reaction shells in distressed concrete were silicified. In later laboratory study (4), chips of deleterious aggregate were silicified in mortar bars. No alkali accompanied the silica introduced from the mortar bar. At this point it was evident that silica present in the reaction shells came from an outside source, and it was postulated (5) that cement paste could be a source of silica because no evidence of the reactivity

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of the fine aggregate was observed during the experiments. The cement paste is no longer considered a source of silica migrating into reaction shells for reasons presented later. The silica is considered to be locally derived from the insoluble residue of the carbonate aggregate itself or possibly from the fine aggregate in the concrete matrix.

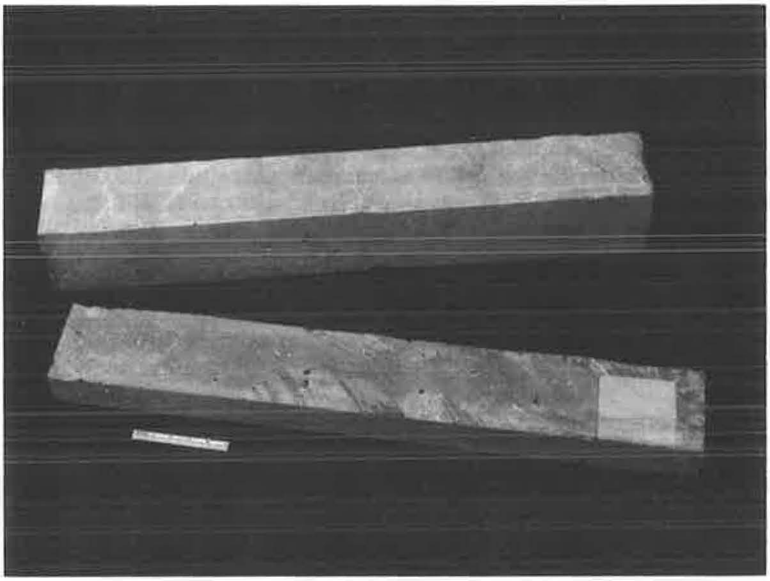

Figure 1. Samples of 2- by 2- by 14-in. cement paste bars: whole bar (upper) and bar cut in half lengthwise showing embedded carbonate aggregate (lower).

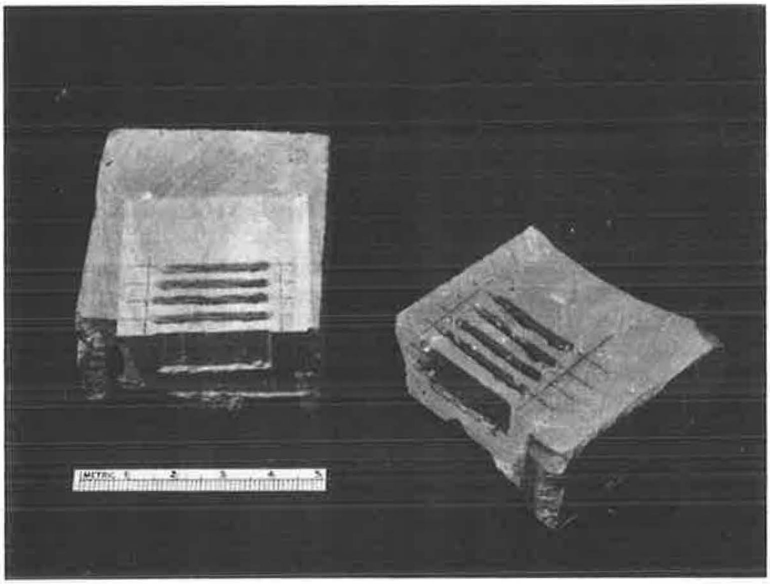

Figure 2. Segments showing spacing of samples in aggregate (left) and in cement paste (right).

The first evidence of the reaction effect on concrete was found when concrete bars made from Glory aggregate and reacted in the laboratory showed no increase in compressive strength (5). Bars made from aggregates with a good service record and reacted at the same time and under the same conditions demonstrated a 50 percent increase in compressive strength.

At this stage of the investigation a more detailed program of research was undertaken with the objective of learning more concerning the nature and mechanism of the reaction. The objective was approached through a study of compositional variations of carbonate aggregates in carbonate aggregate-cement paste reactions.

COMPOSITIONAL VARIATIONS OF CARBONATE AGGREGATES IN CEMENT PASTE

The effect of the reaction on the various components of the aggregate (carbonate minerals, quartz, clay, etc.) was investigated through an approach in which the compositional variations in aggregate and cement resulting from aggregate-cement paste reactions were studied.

Cubes of various carbonate aggregate approximately 1¾ in. on a side were embedded in 2- by 2- by 14-in. bars of cement paste. The cement paste bars were made from Type I cement containing 0.52 percent equivalent Na_2O by mixing 2,500 g of cement with 600 ml of water and curing for 48 hr in the moist room. The bars were then treated by soaking in distilled water at 55 C for a period of 3 mo. After treatment the bars were cut in half lengthwise and samples were taken by means of a dental drill at 5 mm intervals from the cement paste across the interface into the aggregate (Figs. 1 and 2). CaO, MgO, Al_2O_3, and Fe_2O_3 were determined by versene titrametric methods.

TABLE 1

CHEMICAL ANALYSES OF CEMENT PASTE AND ADJACENT AGGREGATES

Specimen	Distance from Interface (mm)	Cement Paste (%)					Distance from Interface (mm)	Carbonate Aggregate (%)				
		SiO_2	CaO	MgO	Fe_2O_3	Al_2O_3		SiO_2	CaO	MgO	Fe_2O_3	Al_2O_3
Newton 19	0	20.80	55.2	7.0	1.27	4.32	0-25	8.55	30.2	21.5	1.10	2.16
	5	19.35	48.4	7.0	2.53	3.06	2.5-5	8.60	29.1	17.9	0.84	1.95
	10	18.90	47.0	7.6	2.27	3.08	5-7.5	13.50	29.7	18.2	0.97	1.84
	15	19.30	47.4	7.7	2.16	3.78	7.5-10	-	28.7	17.8	0.78	2.02
	20	19.25	46.8	8.0	2.13	3.88	10-12.5	10.70	28.13	17.9	0.72	2.16
	40	20.20	50.5	5.4	2.35	4.00	12.5-15	-	29.8	16.7	1.00	1.88
	60	19.85	50.0	6.5	2.48	3.87	15-17.5	-	24.7	19.8	0.35	1.99
							17.5-20	-	28.9	17.8	0.77	1.99
							20-22.5	-	28.6	17.8	0.76	2.10
Newton 6A	0	9.25	30.5	19.4	-	-	0-2.5	10.40	52.4	3.4	-	-
	5	21.60	50.8	2.9	-	-	2.5-5	12.55	52.6	3.2	-	-
	10	18.45	44.9	7.6	-	-	5-8	9.23	49.0	5.7	-	-
	15	18.35	46.1	3.6	-	-	15	10.80	49.6	5.1	-	-
	20	18.15	48.2	5.7	-	-	20	11.20	49.2	5.8	-	-
	40	19.30	48.2	5.6	-	-						
Kenwood C	0	18.5	53.2	2.10	2.94	4.24	0	15.9	34.6	9.70	0.89	1.94
	5	17.6	53.9	2.33	3.00	3.61	5	15.0	36.0	9.60	0.95	1.86
	10	18.2	53.4	2.75	3.16	4.23	15	19.0	31.9	11.30	1.30	1.46
	15	18.1	50.8	4.10	2.60	4.26	20	19.7	30.6	11.60	1.10	2.01
	20	18.4	52.1	3.55	2.66	4.58						
Glory 9	0	17.90	57.4	3.37	-	-	0	8.92	38.1	11.80	-	-
	5	18.95	51.6	5.66	-	-	5	11.02	37.1	11.90	-	-
	10	18.70	51.3	6.24	-	-	10	9.84	35.3	12.90	-	-
	20	18.30	52.9	4.90	-	-	20	10.01	36.0	13.30	-	-
Alite + Glory 11	0	21.7	60.3	0.43	-	-	0	8.96	39.8	10.4	-	-
	5	20.7	61.0	0.76	-	-	5	8.95	38.9	10.1	-	-
	20	20.9	61.0	0.81	-	-	20	9.17	39.2	9.3	0.89	1.13
Belite + Glory 11	0	29.2	55.6	1.00	-	-	0	9.5	33.6	12.4	-	-
	5	29.1	56.2	1.00	-	-	5	9.3	33.2	13.1	-	-
	10	29.1	56.7	0.98	-	-	10	9.2	32.4	14.1	-	-
	15	29.2	56.8	0.95	-	-	15	9.5	32.1	14.5	-	-

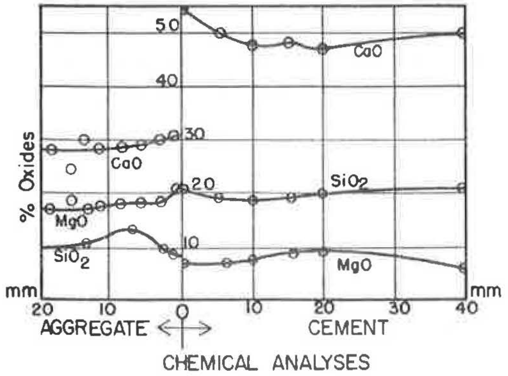

Figure 3. Compositional variations for Newton 19 bar.

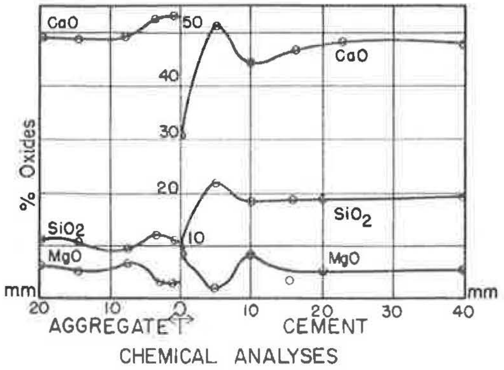

Figure 4. Compositional variations for Newton 6A bar.

Silica was determined by the double dehydration method. Before chemical analyses, semiquantitative X-ray studies were made to determine the mineral or compound content of the aggregate and cement paste, respectively, on the basis of the relative X-ray diffraction peak heights obtained from strip chart recordings of a Norelco X-ray diffractometer. To test the effect of the reaction on the constituents of cement, two cubes of the same reactive aggregate were reacted at pH 12 in bars made from the principal silicate compounds of portland cement, impure alite, and impure belite under the same conditions as the Type I cement paste bars, and sampled accordingly. (The alite and belite were made according to a formula and instructions supplied by L. E. Copeland of PCA Laboratories. A pH 12 environment in the water bath was maintained by adding NaOH solution.)

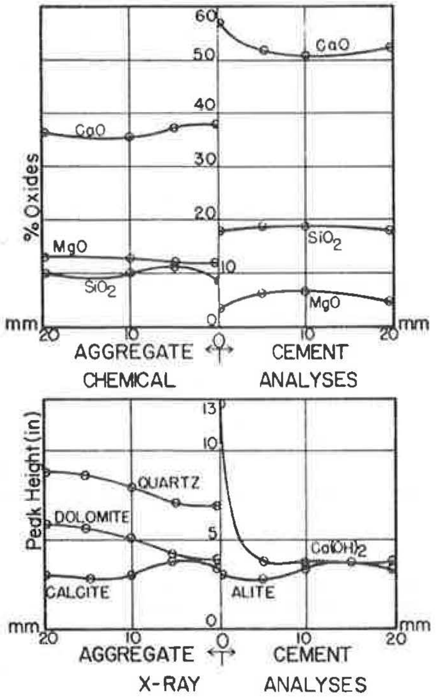

Figure 5. Compositional variations for Glory 9 bar.

The aggregates used were argillaceous dolomitic types which had poor service records or did not pass present acceptance tests of the Iowa State Highway Commission (based essentially on the abrasion loss and durability as related to a water-alcohol freezing and thawing test at Standard Specifications of Iowa State Highway Commission, 1960, pp. 520-521). The Newton 19 and Newton 6A specimens were argillaceous dolomitic limestones from the Coralville member of the Devonian Cedar Valley formation. The Glory 9 and 11 specimens were argillaceous dolomitic limestones from the Rapid member of the Cedar Valley formation. The Kenwood C specimen was an argillaceous calcitic dolomite with a high silica-rich insoluble residue (14 percent) from the Kenwood member of the Devonian Wapsipinicon formation and was the only specimen in which expansion during treatment occurred to the point of cracking the surrounding cement paste.

Analysis of Data

The data are presented in Table 1 and

Figures 3 through 8. Although the experimental approach, nature of the sampling, and procedures employed are such that the study is essentially a reconnaissance approach, the results indicate some significant trends regarding aggregate reactions. The data presented and subsequent interpretations are based on the following assumptions:

1. The aggregate and cement are homogeneous in composition before reaction.
2. The central part of the cubes was least affected by the reaction.
3. The data represent the extent of reaction at the time when the bars were removed from their reaction environment.

The variations in silica content shown in Figs. 3, 4, 6, and 7 are summarized in Fig. 8. In the Newton 19, 6A, and Glory 9 bars (Fig. 8) silica follows a similar pattern in the shell zone interval of 0 to 10 mm from the interface. The silica shows an apparent increase in this zone with the largest amount of silica generally about 5 mm from the interface, whereas at the interface no increase or a slight decrease in silica is indicated. In contrast, the Kenwood C shows a general decrease in silica content throughout the shell zone interval. In the hydrated cement paste, the silica shows variable trends with slight increases at the interface for the Newton 19 and Kenwood C and decrease in the Newton 6A and Glory 9. The Glory 11 aggregate reacted with the hydrated alite and belite (Fig. 7 and lower part of Fig. 8) shows a slight decrease in silica at the interface and a corresponding increase on the cement paste side.

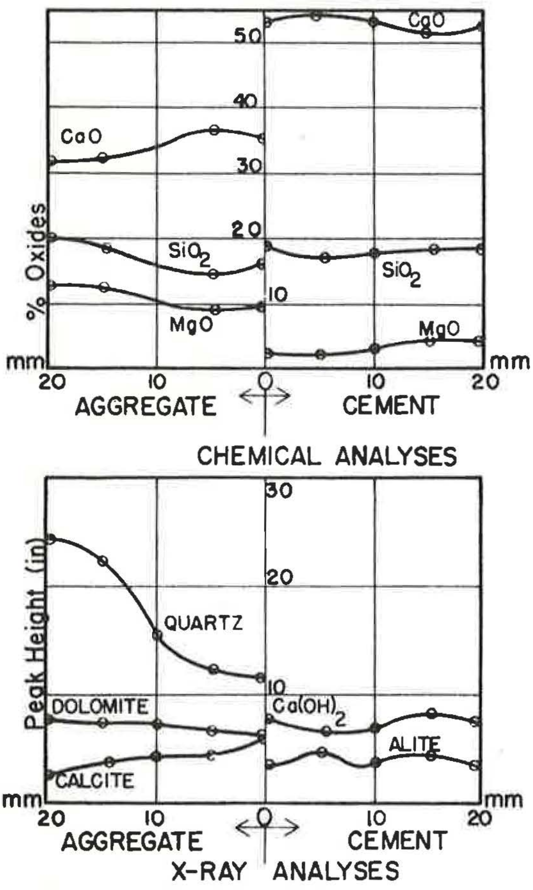

Figure 6. Compositional variations for Kenwood C bar.

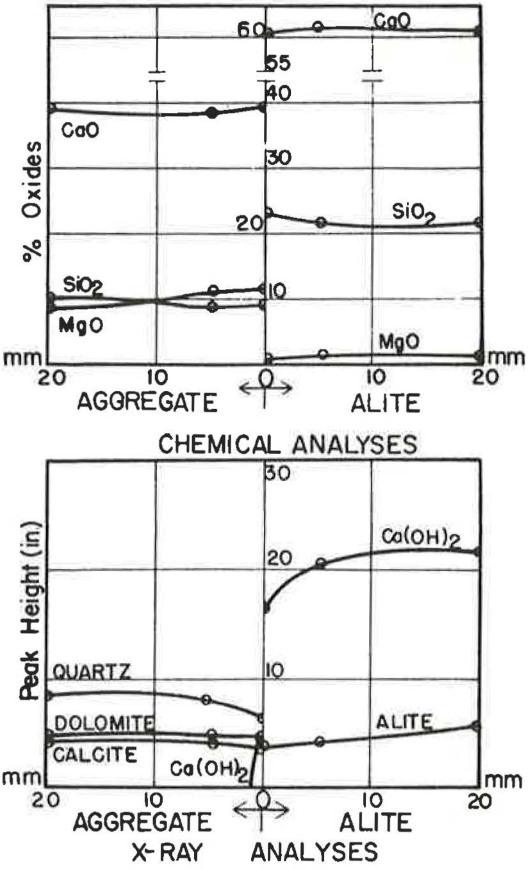

Figure 7. Compositional variations for Glory 11-alite bar.

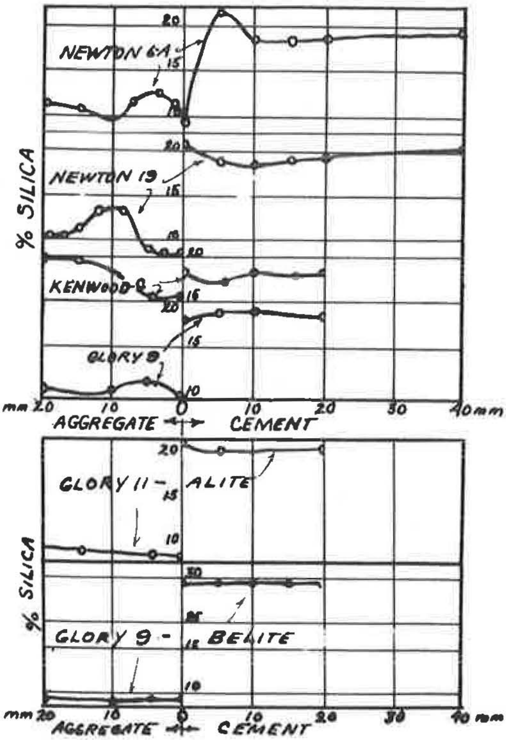

Figure 8. Summary of silica variations.

CaO shows a general increase in reaction zone interval for all the aggregates and variable behavior in the cement paste. MgO in general decreases in aggregates as the interface is approached. Fe_2O_3 and Al_2O_3, where determined, show minor variations and nothing systematic.

The semiquantitative X-ray data in Figs. 5, 6, and 7 show that the quartz content decreases in the reaction zone of the aggregates. Calcite shows an increase in the reaction zone and dolomite a corresponding decrease. The cement compounds represented by hydrated alite are somewhat variable and the X-ray patterns show evidence of some disorder in the cement paste near the aggregate. $Ca(OH)_2$ was found in the Glory 11 aggregate.

Interpretation of Results

The chemical evidence for CaO and MgO and X-ray data for the carbonate minerals show that dolomite appears to be breaking down in the shell zone. This is a concurrent dedolomitization reaction. The calcite increase near the interface may be an apparent effect of the relative decrease in dolomite. It may also reflect formation of some new calcite through the combination of calcium released during dedolomitization or introduced from the cement paste as $Ca(OH)_2$ (Fig. 7), with CO_3 made available through dedolomitization.

The chemical and X-ray data indicate quartz is definitely decreased in the shell zone and must be considered a major source for silica. The quartz is part of the insoluble residue and is probably in a finely divided state. The variable behavior of silica which shows a decrease throughout the reaction zone in the Kenwood C and Glory 11 aggregate and a slight increase at about 5 mm from the interface of the other three aggregates, indicates that local quartz-derived silica can migrate either out of or farther into the rock.

To account for these observations it was initially postulated that silica migration is related to the pH environment and moves in a direction to establish equilibrium. A pH gradient was believed to exist (11) and migration direction of silica will depend on the relative amount of locally available silica on either side of the interface, and movement occurs in an attempt to establish equilibrium.

After this postulation was made additional research brought forth some pertinent

TABLE 2

WATER SOLUBLE COMPONENTS OF CEMENT AFTER ONE YEAR AT 25 C

Alkali Content	Silicon (mg)	Calcium (mg)	Magnesium (mg)	pH
High	0.00	3.34	0.071	11.9
Medium	0.00	4.05	0.062	12.0
Low	0.00	4.53	0.052	11.9

information regarding the pH gradient and cement paste as a silica source. These data supplement the interpretation of the compositional variation data.

Hiltrop (11) demonstrated the presence of a pH gradient across the interface in a relatively simple experiment in which a block of Kenwood C rock 2 by 3½ by 3 in. was embedded within a larger cement paste block so that the rock surface (3 by 3½ in.) was exposed. Holes $^3/_{16}$ in. in diameter were drilled in the rock and cement paste at various distances from the interface. The holes were stoppered with corks and the block was set in distilled water kept at room temperature at such a depth that the water level came within $^1/_8$ in. of the surface of the block. After a period of two weeks water had migrated into the holes in the cement but those in the carbonate rock were dry. The ports were then filled with distilled water and the pH was checked periodically. The equilibrium pH was found to vary from 12 in the cement paste to 9 in the rock at about 5 mm from the interface.

The experiment indicated that a pH gradient exists and demonstrates that hydroxyl ions migrate from the cement paste into the rock because the rock is a poorer source of hydroxyl ions.

Hydrated cement paste was found to be a poor source of silica because of evidence obtained from several areas. Werner (12) determined this by placing 200-g samples of high, medium, and low alkali cements in polyethylene bottles and adding 200 ml of distilled water to each bottle. The samples were allowed to stand at room temperature (23 to 25 C) for one year. The solutions were then analyzed for silicon, calcium, and magnesium, with the results as shown in Table 2.

These additional data indicate that when aggregates are reacted with cement pastes the aggregates are the major source of silica available for migration and the cement paste is the source of the hydroxyl ions which provide the environment for reaction and migration. In re-examining the earlier data on compositional variations, the conditions are such that one would expect silica to migrate from the rock into the cement paste. Such is the case with the Kenwood C and Glory 11. In other aggregates, however, some of the locally derived silica apparently moves farther into the rock leaving a narrow silica deficient zone immediately adjacent to the interface. Migration in this direction, though in response to local conditions, appears to indicate a difference in the adjustment of some rocks to a hydroxyl-rich environment. The complexities of shell formation indicate more research is needed to account for variations in different shells.

An important observation derived from the knowledge of the variable behavior of silica in shell zones is that the presence of a silicified reaction zone in the periphery of an aggregate should not be interpreted as an increase or decrease of the silica content of the shell zone. It does however indicate the presence of either amorphous silica or some silicate compound. Werner's (12) research on these reactions at higher temperatures indicates that mono-calcium silicate hydrates can form as a result of the reaction.

CONCLUSIONS

The compositional variations associated with the carbonate aggregate-cement paste reactions indicate that the reaction is essentially an attempt of the aggregate to come into equilibrium with the higher pH environment produced by the hydrated cement paste. In experiments with cement paste bars, the silica in the shell zones is locally derived from quartz present in the aggregate and it appears to migrate in the direction required to maintain equilibrium according to the local pH environment. Dedolomitization and possibly an increase in new calcite (calcitization) occurs as a concurrent reaction. Cement pastes are stable and are not considered the source of silica found in the shell zone.

The preceding conclusions are valid for the data and conditions described in this report. The conclusions regarding migration of silica from the aggregate into the cement paste are valid only when cement paste is used to provide the reaction environment. Work by Bisque (2) on aggregate chips in portland cement mortar bars and current research indicate conclusively that silica migrates from the mortar into the aggregate.

ACKNOWLEDGMENTS

Most of the data in this paper came from thesis research conducted at Iowa State University and sponsored by the Iowa State Highway Commission. Harwood (10) did the study on compositional variations of carbonate aggregate in cement paste. Hiltrop (11) measured pH changes associated with the reaction, and Werner (12) measured the water soluble components in cement. Lemish directed the research and is responsible for writing this paper.

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Rheology of Fresh Portland Cement Pastes

Influence of Calcium Sulfates

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A study of the influence of the various forms of calcium sulfate on the flow properties of fresh portland cement pastes was made with a coaxial cylinder viscometer. Both calcium sulfate hemihydrate $CaSO_4 \cdot \frac{1}{2}H_2O$ and soluble anhydrite $CaSO_4$ produced high apparent viscosities and yield values in pastes of clinker or quartz. These properties are attributed to the network of gypsum crystals formed from the supersaturated solutions produced by these sulfates. Without the addition of calcium sulfate, the tricalcium aluminates hydrate quickly and the pastes stiffen abnormally.

Shear thickening properties are produced in quartz powder suspensions by both the hemihydrate and the soluble anhydrite. However, a similar trend is not definitely shown by pastes made from clinker and these calcium sulfates. A cement was heated at 120 C to dehydrate the gypsum and a paste of this cement exhibited a shear thickening flow behavior.

• IN A PREVIOUS investigation (1) of the rheology of fresh portland cement pastes, it was demonstrated that pastes usually exhibit first a shear thickening (antithixotropic) and then a shear thinning (thixotropic) flow behavior. The available information (2 through 6) indicated that the calcium sulfates influence the shear thickening properties and under certain conditions cause an abnormal stiffening. This investigation of the influence of the various calcium sulfates on the flow properties of fresh pastes was performed to gain a fundamental understanding of these properties.

In the present investigation the effects of gypsum, calcium sulfate hemihydrate, and the anhydrous forms of calcium sulfate on the flow properties of suspensions of quartz powders and of portland cement were studied with a coaxial cylinder viscometer. Although the fundamental natures of the flow properties of concentrated suspensions and gels are not yet completely understood, it is possible by the techniques and theories of rheology and physical chemistry to obtain a great deal of information about the factors that influence the flow properties of these complicated systems. Eventually by performing careful experimental and theoretical studies, a fundamental understanding of the mechanism of the flow of concentrated suspensions and gels will result.

The nomenclature for the flow properties will follow the recommendations of the Committee on Communication Problems in Rheology (7). The recommended nomenclature is based on the measurements; i.e., the shearing stress S, the rate of shear D, and time of shear t. When the ratio S/D, at a particular rate of shear, increases with time of shear, the system is called shear thickening. The ratio S/D is defined here as the apparent viscosity at a certain rate of shear. This type of behavior is exhibited (Fig. 1) by some cement pastes 20 min after the cements are placed in contact with water. A shear thinning system is exhibited by a decrease in the S/D values with time of shear at a particular rate of shear. A shear thinning behavior may be seen in Figure 1 for a cement paste 185 min after the cement was placed in contact with water. When the S/D values remain constant with time of shear the system is termed time-independent. Time-dependent effects in S/D values indicate that changes in the structure of the system are proceeding.

Paper sponsored by committee on Basic Research Pertaining to Portland Cement and Concrete.

It is well to review some of the chemical reactions that occur when cement is mixed with water. C_3S, the principal component of cement, hydrates continuously after it is brought in contact with water. The hydration of C_3S leads to a shear thinning flow behavior and a gradual increase in the apparent viscosities of the pastes (1). Because C_2S hydrates slowly, this component does not contribute markedly to the flow properties of the fresh pastes. Unless the hydration of C_3A and C_4AF (8) is retarded, the pastes stiffen very rapidly producing a condition called quick set. According to present information (1), the products of the reaction of C_3A lead to shear thinning flow properties and a marked increase in the apparent viscosities of the pastes. The influence of the sulfoaluminates on the flow properties has not been thoroughly investigated. However, it has been reported by Budnikov (9) that the sulfoaluminates cause high early strength in concrete.

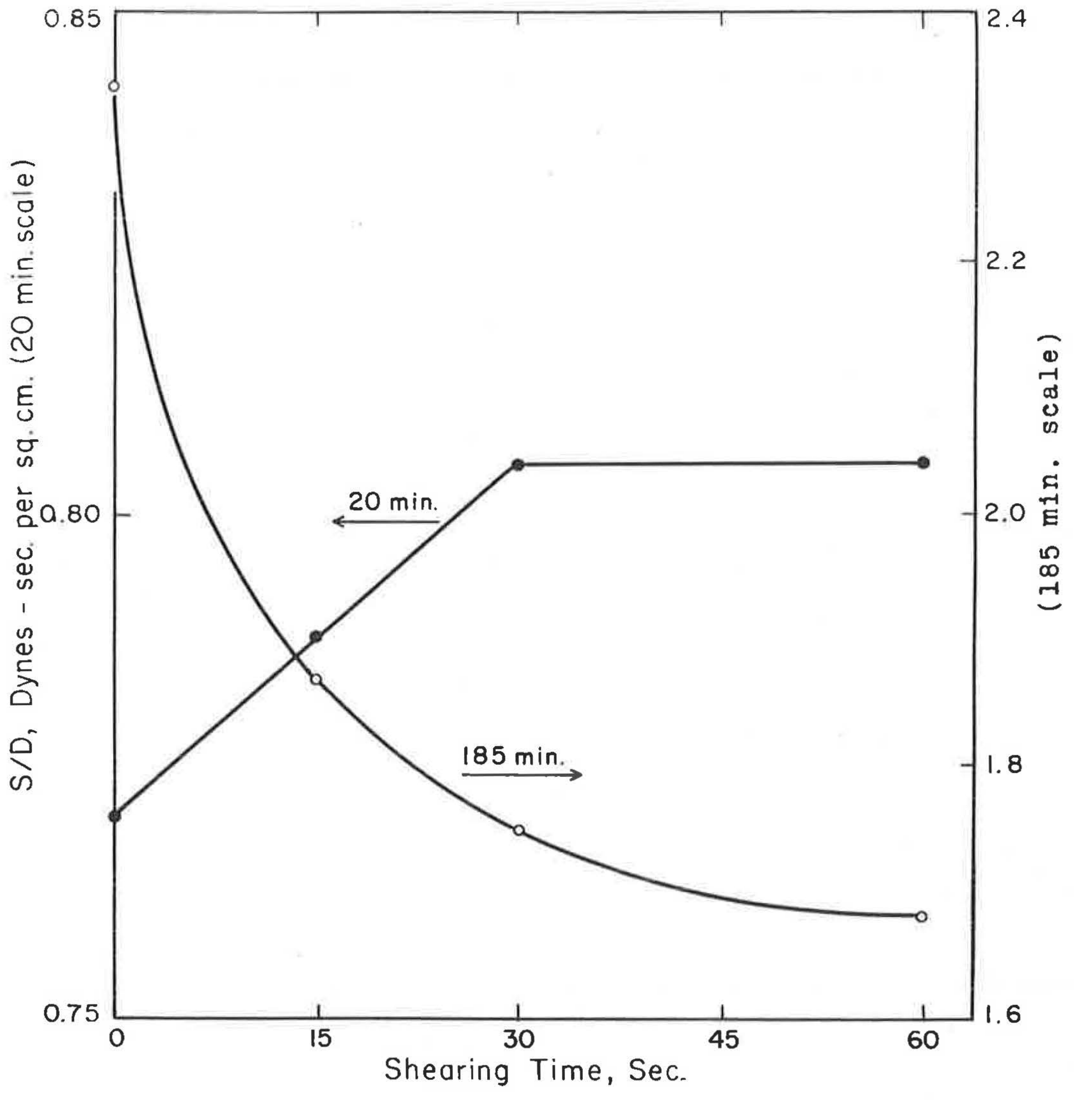

Figure 1. S/D values as a function of shearing time for cement 15366 at 20- and 185-min hydration times ($D = 658\ sec^{-1}$).

Under normal conditions the C_3A dissolves (Fig. 2) to form solutions of calcium and hydroxyl ions and aluminic acid and its ions. In the absence of calcium sulfate, C_4AH_{19} is formed (10). When sufficient hydrated calcium aluminate is produced, a rapid change in flow properties results. This is accompanied by the evolution of a large amount of heat. In the presence of calcium sulfate the high sulfate sulfoaluminate, ettringite, $C_3A \cdot 3CaSO_4 \cdot 31H_2O$, is formed. A coating of the sulfoaluminates on the C_3A particles is considered the cause of the decrease in the rate of C_3A hydration (2).

When gypsum $CaSO_4 \cdot 2H_2O$ is ground with clinkers (Fig. 2), some of it is dehydrated to the hemihydrate $CaSO_4 \cdot \frac{1}{2}H_2O$ and soluble anhydrite $CaSO_4$ (11). Hemihydrate is formed when the soluble anhydrite is exposed to the atmosphere. Because the hemihydrate and the soluble anhydrite are much more soluble than gypsum, the solutions in contact with these substances are supersaturated with respect to gypsum. Crystallization of gypsum will therefore proceed under the proper conditions.

Evidence for the dehydration of gypsum was found in an analysis (12) of the compositions of the aqueous phases in contact with cements. Solutions supersaturated with respect to gypsum exist for at least 30 min after the cements are mixed with water. The concentration of calcium sulfate in solution may be reduced by the formation of sulfoaluminates or by the crystallization of gypsum.

Bucchi (13) reported that a large fraction of the sulfate reacts to form sulfoaluminate in the first few minutes after mixing. It was demonstrated by him that the sulfate is removed at rates proportional to the aluminate content.

Solutions supersaturated with respect to gypsum are unstable and, in the presence of nucleating agents, a rapid crystallization of gypsum occurs. Under the proper conditions a network of gypsum crystals may result. Both gypsum and clinker may act as nucleating agents according to Manabe (6). However, exposure of the cement to the atmosphere may cause a deactivation of the clinker nuclei which could delay the crystallization process for some time after the cement had been mixed with water.

The network of gypsum crystals that is deposited from supersaturated calcium sulfate solutions causes a change in flow properties which is sometimes called plaster set. In the present study, plaster set will also refer to the network of gypsum crystals that produces the set. There was some experimental evidence that seemed to relate the plaster set to the shear thickening properties of fresh pastes:

1. The shear thickening properties are evident 15 min after the cement is mixed

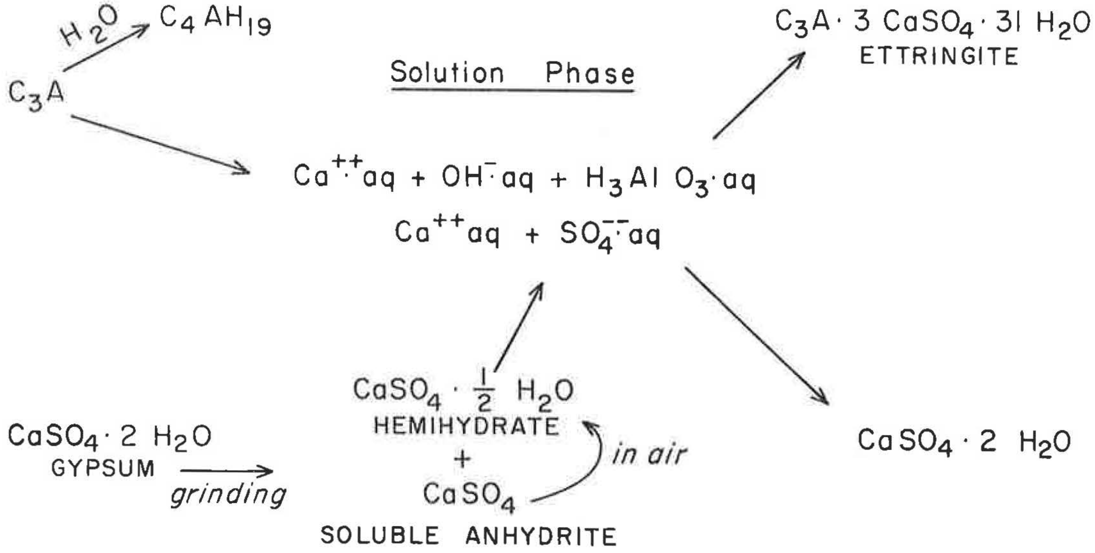

Figure 2. Chemical processes in hydration of C_3A and calcium sulfates.

with water. After an hour or two this behavior is no longer found. Plaster set under normal conditions would be expected almost immediately after the cement is mixed with water and the network of gypsum crystals should not dissolve completely before an hour or two have elapsed (2).

Such hydration products as the hydrated calcium silicates and aluminates produce an increase in the apparent viscosities, S/D, of the cement pastes and in the shear thinning behavior. Therefore, as the cement continues to hydrate, there will be an increase both in the apparent viscosity and in the shear thinning. Consequently, the shear thickening behavior would become less evident as the gypsum in the plaster set dissolves, and the hydration of the clinker proceeds.

2. Gypsum suspensions have been reported to exhibit a shear thickening behavior (14).

3. Suspensions of attapulgite crystals have been reported by Gabrysh, Ree, Eyring, McKee, and Cutler (15) to exhibit shear thickening properties similar to those found in cement pastes. Attapulgite exists as a network of needlelike crystals similar to that found with secondary gypsum crystals. The increase in apparent viscosity with time of shear has been attributed by these authors to the higher viscosity of the system when the networks of crystals are broken up by shearing action. It is reasonable to assume that a suspension of individual needle-shaped crystals would exhibit a higher apparent viscosity than aggregates of these crystals.

On the other hand, Dellyes (5), who first reported the shear thickening properties of freshly mixed pastes, showed that carbonates and carbonated pastes exhibit shear thickening flow behaviors. Recently it was reported (16) that calcium carbonates may cause some suspensions of silicates to show a shear thickening behavior.

One type of abnormal stiffening of portland cement pastes, mortars, and concretes is defined by committees of the American Society for Testing Materials as false set (4). This behavior is attributed to a plaster set. When this stiffening occurs, not much heat is evolved. The present paper deals with plaster set at hydration times longer than those employed in the ASTM false set test. The extension of the rheological investigation to shorter hydration times and the correlation with ASTM false set test results are left for future work.

TABLE 1

POTENTIAL COMPOUND COMPOSITIONS

Type of Component	Percent of Component							
	Type I Cement 15366	High Alkali 15698	Clinker 15367	LTS No. 18	Medium C_3A 15622A	High C_3S 15497B	Low C_3A 15669	Zero C_3A 19648
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
C_3S	44.8	45.3	45.4	44.5	49.2	60.1	33.0	66.0
C_2S	26.9	28.6	28.5	28.0	28.5	11.9	54.2	14.7
C_3S/C_2S	1.67	1.58	1.59	1.59	1.73	5.05	0.61	4.50
C_4AF	6.7	7.6	6.8	6.8	12.8	7.9	5.8	10.15[a]
C_3A	13.6	9.8	14.3	13.2	4.4	10.3	2.3	0.0
$CaSO_4$	3.3	3.0	-	3.1	2.7	3.1	3.1	4.3
MgO	2.5	3.4	2.63	2.6	1.44	1.72	1.72	0.60
Free CaO	0.93	0.08	0.98	0.3	0.49	3.14	0.15	0.58
Total alkali ($Na_2O + K_2O$)	0.33	1.57	0.33	0.25	0.22	0.70	0.27	0.12
Surface area (sq cm/g) (Wagner)	2,260	2,250	2,250	1,750	2,200	2,500	2,280	2,640

[a] Contains 3.16 percent C_2F.

RHEOLOGICAL EQUIPMENT

For this investigation a Haake Rotovisco coaxial cylinder viscometer (Brinkmann Instruments, Inc., Great Neck, N.Y.) was employed. The radius of the MVI bob is 2.003 cm and that of the cup is 2.102 cm. The constants of the instrument were listed in a previous paper (1). The bob was rotated at speeds up to 580 rpm. Torques were measured by the displacement of springs, with three different ranges up to a shearing stress of 3,000 dynes per sq cm. By placing carbon powder on the top of a suspension in the cup and rotating the bob, it was possible to observe that the entire sample was flowing. The entire sample did not flow when wider annular spaces were used.

MATERIALS

The following portland cements were examined: Type I cement 15366, high alkali 15698, Long Time Study No. 18, medium C_3A 15622A, high C_3S 15497B, low C_3A 15669, and a zero C_3A 19648. A clinker 15367 from the batch used to make cement 15366 was ground in a ball mill to a Wagner surface area (17) of 2,200 sq cm per g. The potential compositions of these materials are given in Table 1. The SO_3 content of the clinker is 0.41 percent. Cements 15366, 15622A, 15497B, 15669 and 19648, as well as clinker 15367 were especially prepared in commercial plants for this laboratory. The conditions of preparation were those normally used in the plants. Extensive examinations of the chemistry and properties of these cements have been made in this laboratory.

The quartz powders (Silver Bond B Grade, Tamm Industries, Chicago, Ill.) exhibited a particle size distribution similar to that of the cement 15366 with a Wagner surface area of 2,260 sq cm per g (18). All the powder passed through a 325 mesh screen.

Baker reagent grade calcium sulfate hydrate, $CaSO_4 \cdot 2H_2O$, was the source of all the calcium sulfates investigated. The X-ray diffraction pattern of the dihydrate was typical for this substance. However, low intensity peaks of the hemihydrate diffraction pattern were found. The ignition loss of 19.7 percent (th. 21.0 percent) corresponds to 8.1 percent hemihydrate.

The procedures for the preparation of the hemihydrate and anhydrous sulfates followed those of Ljunggren (19). The calcium sulfate hemihydrate, $CaSO_4 \cdot \frac{1}{2}H_2O$, was prepared by heating the dihydrate for one day at 120 C. The heated sample was then allowed to cool exposed to the air. Under these conditions any soluble anhydrite formed at 120 C would be rehydrated to $CaSO_4 \cdot \frac{1}{2}H_2O$. The hemihydrate was identified by X-ray diffraction and weight loss on ignition at 1,050 C. The soluble anhydrous calcium sulfate, α-$CaSO_4$, was made by dehydrating the dihydrate for one day at 200 C. This sample was allowed to cool in a dessicator. The weight loss of the sample was negligible at 1,050 C. The insoluble anhydrite, β-$CaSO_4$, was prepared by igniting the

TABLE 2

EFFECT OF REPEATED SHEAR STRESS ON RHEOLOGICAL BEHAVIOR OF FRESH CEMENT PASTE

Hydration Time (min)	Shearing Time (sec)	S/D (dynes-sec/sq cm)[a]	$\frac{\Delta(S/D)}{S/D}$
(1)	(2)	(3)	(4)
20	0	0.77	-
	15	0.79	+0.026
	30	0.81	+0.050
	60	0.81	+0.050
185	0	2.34	-
	15	1.87	-0.22
	30	1.75	-0.29
	60	1.68	-0.33

[a] D = 658 sec^{-1}.

dihydrate at 500 C for one day. No appreciable ignition loss was exhibited by this sample at 1,050 C.

PROCEDURES

In general, the procedures described in a previous study (1) were employed. However, to reduce the amount of the solids required, the method for mixing the solids and water was revised. In only a few experiments was the mixing performed in a Waring Blendor under vacuum. In the present study it was found more convenient to mix samples in a glass bottle of 120-ml capacity on a Spex Shaker (Scotch Plains, N.J.). Only about 100 g of solids are necessary in this procedure. The results obtained with samples mixed on the Spex Shaker were almost the same as those obtained with the samples mixed in the Waring Blendor.

In some experiments, to prevent the mixtures from reaching temperatures excessively above 25 C, ice was added to the mix water and included in the total weight of water. In most cases the samples were mixed for 5 min to a final temperature of approximately 25 C. The samples after mixing were placed in a 25 C constant temperature bath for 15 min. They were then inserted into the viscometer cups and the rpm-torque measurements were made.

The volume concentrations of the quartz in the suspensions were maintained constant at 43 volume percent. This volume concentration is produced by a 0.5 water to quartz ratio by weight. This weight concentration (w/c ratio of 0.5) provides 39 volume percent concentration in the case of the cement experiments. Deviations from this volume percent of solids are pointed out later.

The procedures for making the viscometer measurements were described previously (1) and are essentially the same as those recommended by Green (20). An upcurve was made which relates the rpm and torque values for increasing rpm values, and immediately afterwards a downcurve was made for decreasing rpm values. The two curves are called a flow curve. In each experiment two flow curves were made with the second curve following immediately after the first. Each flow curve corresponds to one cycle and requires about 3 min to make.

The ratio of shearing stress S to rate of shear D at a rate of shear 658 sec^{-1} is defined as the apparent viscosity and is given in dynes-seconds per square centimeter. The S/D values are taken at a rate of shear of 658 sec^{-1}. The second rheological parameter evaluated, is the yield stress f (20, 21). Theoretically the yield stress is the minimum shearing stress necessary to initiate the flow of a plastic material. The

TABLE 3

GYPSUM MIXTURES, INFLUENCE OF HEMIHYDRATE ON FLOW PROPERTIES OF QUARTZ-WATER SYSTEM

Hemihydrate (% total $CaSO_4$)	1st Cycle		2nd Cycle		Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	
(1)	(2)	(3)	(4)	(5)	(6)
0	0	0.20	0	0.19	-0.051
20	5	0.22	10	0.22	0
40	10	0.24	10	0.24	0
50	10	0.27	15	0.27	0
60	40	0.34	50	0.37	+0.085
80	80	0.49	100	0.53	+0.078
100	160	0.69	180	0.76	+0.097

yield stress in this investigation is determined by the extrapolation of the straight portion of the downcurve to the torque axis. The intercept in torque units is converted to the yield stress by the appropriate constants (1).

In the tables the yield stresses f in dynes per square centimeter and apparent viscosities, S/D, in dynes-seconds per square centimeter are listed for the first and second cycles. In the final column the over-all behavior is characterized by a term Δ(S/D)/(S/D). The Δ(S/D) values are the changes in apparent viscosities between the first and second cycles. When Δ(S/D) is divided by the average of the S/D values in the first and second cycles, the Δ(S/D) values are reduced to a proper order of magnitude when there is a large variation in S/D values in a set of experiments. Positive, negative, and zero values of Δ(S/D)/(S/D) correspond to shear thickening, shear thinning, and time-independent systems, respectively. The values of Δ(S/D)/(S/D) correspond to some extent to the degree of shear thickening or thinning behavior. Values of Δ(S/D)/(S/D) ranging between 0 and ±0.02 are within experimental error, but are listed because in some cases trends in the values may be noted.

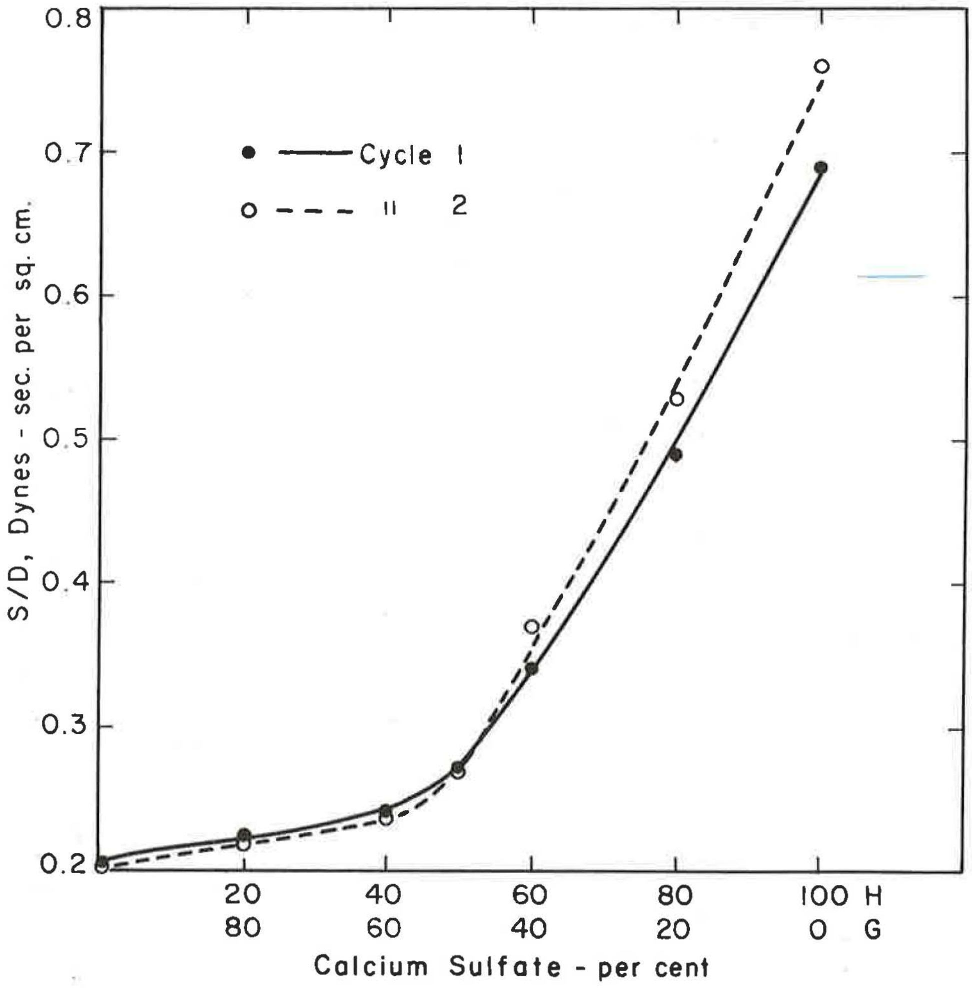

Figure 3. Influence of hemihydrate-gypsum mixtures on S-D values of quartz-water system.

RESULTS

Changes in Flow Properties with Hydration Time

The apparent viscosities of cement 15366 paste with a w/c = 0.5 were determined as a function of shearing time at two hydration periods. The determinations were made on separate samples 20 and 185 min after the cements were placed in contact with water. Table 2 gives the apparent viscosities at 0, 15, 30, and 60 sec of shearing time. In Figure 1 it may be seen that at 20 min of hydration time the S/D values increase from 0.77 to 0.81 dynes-sec per sq cm with time of shear to a constant value at 30 sec. After 60 sec of shearing time the S/D values begin to decrease. A decrease in S/D values with time of shear from 2.34 to 1.68 dynes-sec per sq cm may be noted at 185 min of hydration time. The sample at 20-min hydration time illustrates a shear thickening material, whereas the sample at 185 min of hydration time demonstrates a shear thinning material.

Relationships Between the Flow Behaviors and Calcium Sulfates

Three sets of experiments were conducted to show the influences of the various forms of calcium sulfate on the flow behaviors of fresh cement pastes and quartz suspensions:

1. The effects of the calcium sulfates on the flow properties of quartz powder suspensions were examined. Quartz suspensions were used previously by Lerch and Bogue (22) to demonstrate the stiffening caused by the hemihydrate.

TABLE 4

EFFECT OF CALCIUM SULFATE ON RHEOLOGICAL BEHAVIOR OF QUARTZ-WATER SYSTEM

Type of Calcium Sulfate	Sulfate Solid Wt. (%)	1st Cycle		2nd Cycle		Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
		Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	
	(1)	(2)	(3)	(4)	(5)	(6)
Gypsium	1½	0	0.19	0	0.19	0
	2	0	0.19	0	0.19	0
	3	0	0.20	0	0.19	-0.051
	5	0	0.20	0	0.20	0
	7	10	0.22	0	0.22	0
Hemihydrate	1½	40	0.37	40	0.39	+0.053
	2	50	0.42	60	0.46	+0.091
	3	160	0.69	180	0.76	+0.097
	5	500	1.60	520	1.72	+0.072
	7	1,000	2.83	1,010	2.97	+0.048
Anhydrite: soluble	1½	40	0.38	45	0.40	+0.026
	2	60	0.44	60	0.47	+0.066
	3	200	0.82	210	0.95	+0.147
	5	570	1.88	580	1.98	+0.052
	7	1,080	3.24	1,050	3.25	+0.003
Insoluble	1½	5	0.21	10	0.21	0
	2	5	0.22	5	0.22	0
	3	10	0.22	10	0.23	+0.044
	5	10	0.25	10	0.25	0
	7	15	0.28	15	0.28	0

2. An investigation was made of the effects of the various forms of calcium sulfate on the flow properties of ground clinker pastes.

3. The effects of heating a cement to 120 C on the flow behavior of the paste were studied.

In the first set of experiments mixtures of hemihydrate and gypsum were added to 43 volume percent quartz powder suspensions. In each case the total $CaSO_4$ content was 3 percent by weight of the quartz. The percentages of hemihydrate ranged from 0 to 100 percent. The weights of the quartz in the mixture were 100 g minus the weight of the calcium sulfates. Inasmuch as the densities of quartz, gypsum, hemihydrate, soluble anhydrite, and insoluble anhydrite are 2.65, 2.32, 2.35, 2.45, and 2.96, respectively, the volume percents of solids are slightly different from 43 volume percent. This small variation in volume percent of solids would not be expected to cause a marked change in the yield values and apparent viscosities. The results of these experiments are given in Table 3 and Figure 3. Table 3 (col. 1) gives the percent of hemihydrate as $CaSO_4$. The yield values f and apparent viscosities S/D at 658 sec^{-1} are given in columns 2 and 3, and 4 and 5 for the first and second flow cycles, respectively. The over-all behavior is demonstrated by the $\Delta(S/D)/(S/D)$ values in column 6.

Figure 3 shows the S/D values in the first and second cycles as a function of the hemihydrate content. At about a 50 percent hemihydrate content, the S/D values begin to increase sharply. Also, the first and second cycle curves cross at this point. The lower values of S/D in the second cycle up to a 50 percent concentration of hemihydrate demonstrate that the systems are shear thinning in this range. The shear thickening properties of the suspensions above a 50 percent hemihydrate are demonstrated by the higher S/D values in the second cycle. In Table 3, column 2, a rise may be seen in yield stresses from 0 to 160 dynes per sq cm with a hemihydrate increase from 0 to 100 percent. The sharpest increase in yield values begins at a 50 percent hemihydrate content. The over-all character of the flow behavior is demonstrated by the changes in $\Delta(S/D)/(S/D)$ values (column 6) from -0.051 to +0.097 with the increase in hemihydrate content from 0 to 100 percent. The negative values correspond to a shear thinning system, the zero values to a time-independent system and the positive values to a shear thickening system. Thus with an increase in the hemihydrate content any of these three kinds of flow behaviors may be observed.

The effects of gypsum, hemihydrate, soluble anhydrite, and insoluble anhydrite on the flow properties of the 43 volume percent quartz-water system are shown by the

TABLE 5

INFLUENCE OF HEMIHYDRATE-GYPSUM MIXTURES ON FLOW PROPERTIES OF A CLINKER-WATER SYSTEM

Hemihydrate (% total $CaSO_4$)	1st Cycle		2nd Cycle		Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	
(1)	(2)	(3)	(4)	(5)	(6)
0	200	0.51	200	0.52	+0.019
20	190	0.49	190	0.50	+0.020
40	110	0.34	110	0.35	+0.029
50	120	0.36	120	0.36	0
60	110	0.34	110	0.34	0
70	170	0.47	170	0.48	+0.021
80	195	0.53	190	0.54	+0.019
100	270	0.70	270	0.69	-0.014

data in Table 4. The amounts of sulfate solids added are listed in column 1 in weight percent of the quartz. In each experiment the total weight of the solids was held constant at 100 g. Both hemihydrate and soluble anhydrite produced shear thickening flow behaviors (col. 6). The Δ(S/D)/(S/D) values for the suspensions with these two forms of calcium sulfate do not show marked differences. The Δ(S/D)/(S/D) values increase to a maximum at 3 percent and then decrease with an increasing sulfate content. However, the yield values in the first cycle of the suspensions with hemihydrate and soluble anhydrite additions increase to about 1,000 with sulfate additions up to 7 percent. An increase in apparent viscosities from 0.37 and 0.38 to 2.83 and 3.24 dynes-sec per sq cm is found with hemihydrate and soluble anhydrite, respectively.

In general, gypsum and insoluble anhydrite produced time-independent flow behaviors in quartz suspensions. The yield values for these suspensions were less than 15 dynes per sq cm. Both gypsum and insoluble anhydrite produced relatively small effects on the yield values and apparent viscosities, but the effect of insoluble anhydrite was slightly greater.

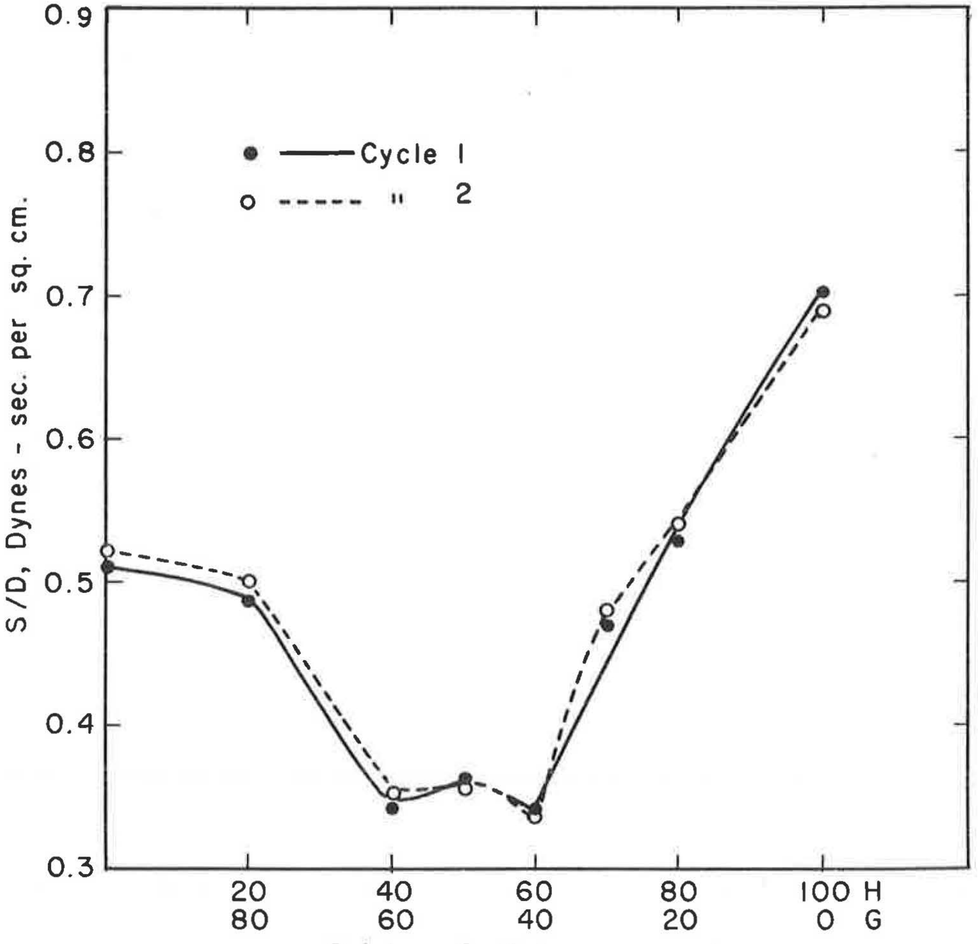

Figure 4. Influence of hemihydrate-gypsum mixtures on S/D values of clinker-water system.

In the second group of experiments the effects of hemihydrate-gypsum mixtures on the flow behavior of ground clinker (15367) pastes with a w/c ratio equal to 0.5 were investigated. The $CaSO_4$ contents of the additions were maintained constant at 3 percent by weight of the clinker. The hemihydrate contents of the mixtures ranged from 0 to 100 percent by weight of $CaSO_4$. The results are summarized in Table 5 and Figure 4. In general, the $\Delta(S/D)/(S/D)$ values (col. 6) indicate that the systems are slightly shear thickening. It is necessary to point out, however, that most of the values are in the range 0 ± 0.02, which is within experimental error. In Figure 4, the S/D ratios for the first cycle show a decrease in values from 0.51 to 0.34 dynes-sec per sq cm in the range of hemihydrate contents 0 to 40 percent. In the range 60 to 100 percent hemihydrate, the values increase sharply from 0.34 to 0.70 dynes-sec per sq cm. In Table 5 (col. 2), the yield values in the first cycle decrease from 200 to 110 dynes per sq cm with an increase in hemihydrate content from 0 to 40 percent. In the range 60 to 100 percent hemihydrate, the yield values increase sharply from 110 to 270 dynes per sq cm.

A hemihydrate content of 60 percent is optimum in these experiments for reducing the yield values and apparent viscosities to a minimum. Later, these effects are shown to be based on the rates of solution of the calcium sulfates, and the rates of reaction of C_3A.

Table 6 gives the results of a series of experiments to establish the effects of gypsum, hemihydrate, soluble anhydrite, and insoluble anhydrite on the flow properties of clinker pastes. The 1.5 to 7 percent calcium sulfate additions are given in column 1 in percent by weight of the solids. In each experiment the w/c is 0.5 with the calcium

TABLE 6

INFLUENCE OF CALCIUM SULFATE ON RHEOLOGICAL PROPERTIES OF A CLINKER-WATER SYSTEM

Type of Calcium Sulfate	Sulfate Solid Wt. (%)	1st Cycle		2nd Cycle		Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
		Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	
	(1)	(2)	(3)	(4)	(5)	(6)
Gypsium	2½	175	0.47	180	0.47	0
	3	200	0.51	200	0.52	+0.019
	5	130	0.38	130	0.38	0
	7	120	0.42	120	0.43	+0.024
Hemihydrate	1½	140	0.39	140	0.40	+0.025
	2	170	0.45	165	0.46	+0.022
	3	270	0.70	270	0.69	-0.014
	5	290	0.73	300	0.74	+0.014
	7	410	1.00	400	1.00	0
Anhydrite:	1½	120	0.35	120	0.36	+0.028
soluble	2	150	0.43	150	0.44	+0.023
	3	245	0.61	250	0.62	+0.016
	5	330	0.80	340	0.81	+0.012
	7	410	1.03	400	1.01	-0.020
Insoluble	1½	710	1.60	580	1.28	-0.222
	2	1,010	2.02	960	1.91	-0.056
	3	910	1.88	900	1.85	-0.016
	5	1,060	2.15	1,020	2.09	-0.028
	7	1,010	2.07	960	2.00	-0.034

sulfates included in the weight of solids. Pastes with hemihydrate and soluble anhydrite showed a tendency to be slightly shear thickening. Most of the Δ(S/D)/(S/D) values (col. 6) were small enough to be in the range of experimental error. Also, no clear correlation between these values and the sulfate contents was apparent; if anything, there is a slight decrease in the Δ(S/D)/(S/D) values with sulfate content. On the other hand, yield stresses and S/D values increase markedly with sulfate content. The pastes with hemihydrate exhibited, in the first cycles, a rise in yield value from 140 to 410 dynes per sq cm in the sulfate content range 1.5 to 7 percent. In the same range of sulfate contents the S/D values rose from 0.39 to 1.00 dynes-sec per sq cm. For the same additions of soluble anhydrite the yield values rose from 120 to 410 dynes per sq cm and the S/D values increased from 0.35 to 1.03 dynes-sec per sq cm.

The addition of gypsum produced some indication of a slight shear thickening behavior at 3 and 7 percent. Small changes in yield stress and S/D values were observed, and in general there was a decrease in the values with sulfate content. On the other hand, the suspensions with insoluble anhydrite additions exhibited a marked shear thinning behavior. The Δ(S/D)/(S/D) value (col. 6) was largest for the 1.5 percent addition. The yield values in the first cycle ranged between 710 and 1,060 dynes per sq cm, which are more than double those of the pastes with the other forms of sulfate. The apparent viscosities were between 1.60 and 2.07 dynes-sec per sq cm. The high yield stresses and S/D values in this set of experiments may be attributed to the rapid hydration of C_3A in the presence of a sulfate that dissolves slowly. These relationships are discussed later.

A control paste without any sulfate added was too stiff to measure, but a shear thinning behavior was observed.

In the third set of experiments a cement was heated at 120 C to dehydrate the gypsum. The flow properties of pastes of this cement were compared to those of pastes of the unheated cement. The Long Time Study No. 18 cement was used for this set of experiments. The procedure of heating the cement for 18 hr at 120 C has been reported to encourage abnormal stiffening. Two flow curves were made at 20, 50, 125, and 185 min after the cements were added to the water. The results are summarized in Table 7 and Figure 5.

TABLE 7

EFFECTS OF HEATING AN LTS 18 CEMENT AT 120 C ON RHEOLOGICAL PROPERTIES

Hydration Time (min)	1st Cycle		2nd Cycle		Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	
		Unheated			
(1)	(2)	(3)	(4)	(5)	(6)
20	60	0.23	60	0.24	+0.043
50	60	0.23	60	0.23	0
125	110	0.37	110	0.37	0
185	170	0.55	170	0.53	-0.037
		Heated			
20	160	0.46	165	0.47	+0.022
50	160	0.45	165	0.47	+0.044
125	300	0.78	300	0.78	0
185	375	0.92	340	0.88	-0.044

In Figure 5 the relationships are shown between the S/D values for two cycles for the heated and unheated cements and the hydration times. The pastes with heated cements exhibit S/D values almost twice those of the pastes with unheated cement. The values are almost constant between 20 and 50 min and then begin to increase rapidly. The yield values of the pastes with unheated cement are only about one-half those of the pastes with the heated cement (cols. 2 and 4). The Δ(S/D)/(S/D) values (col. 6) showed in both sets of experiments that the systems changed with hydration time from shear thickening to shear thinning. The shear thickening behavior of the paste of the heated cement, as indicated by the Δ(S/D)/(S/D) values, was evident even at 50 min of hydration

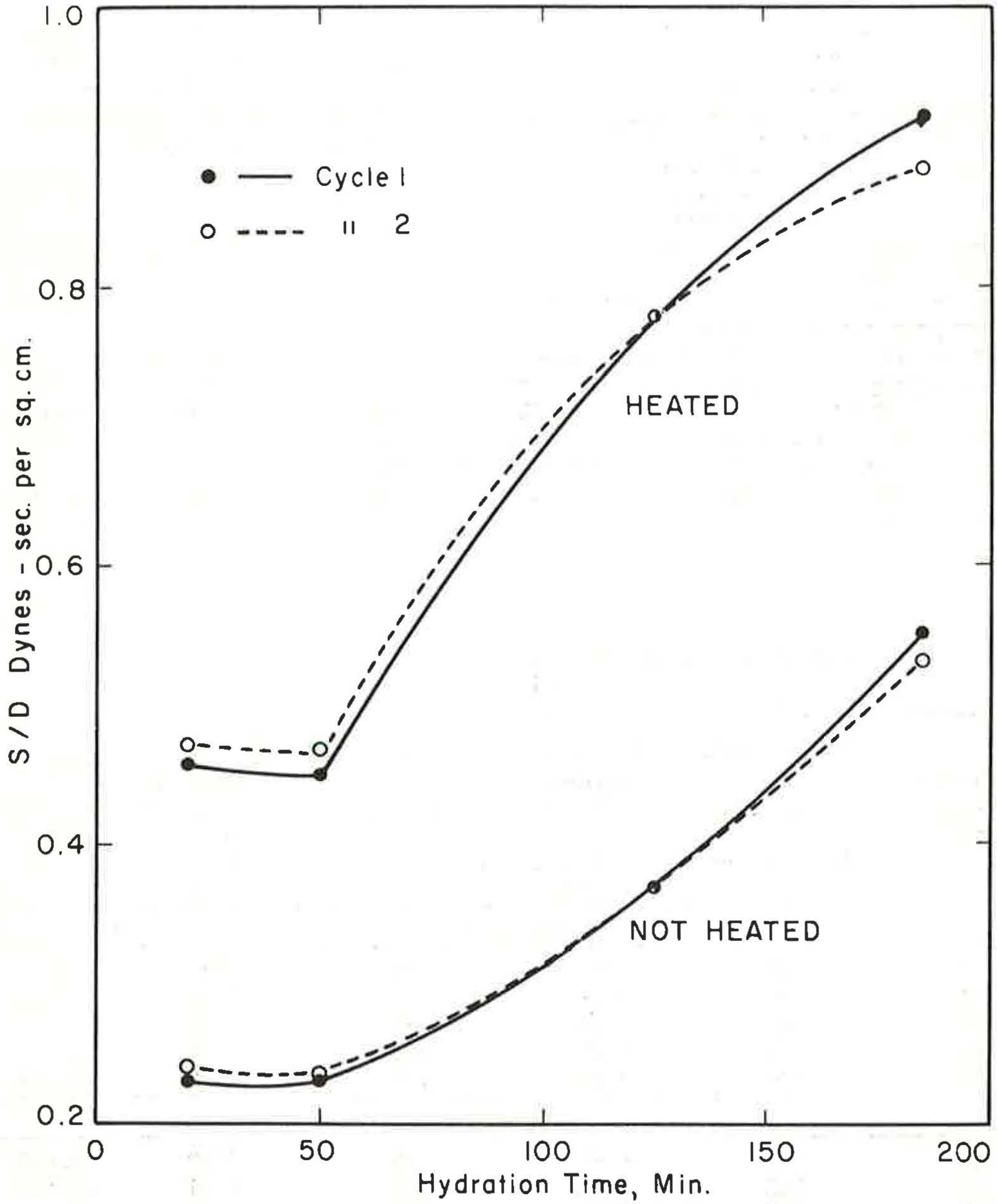

Figure 5. Effect of heating a cement at 120 C on S/D values as a function of hydration time.

time, whereas the paste of the unheated cement exhibited a time-independent behavior at this hydration time.

Mixing Time and Temperature

The effect of mixing time on the flow properties of cement 15366 was investigated. Because mixing exerts a heating effect, it was not possible, even with ice, to hold the temperature after mixing at 25 C beyond a 5-min period of mixing. To study the influence of temperature, two sets of experiments were performed. In one set of experiments no ice was added to the mixtures. The results of these experiments are given in Table 8. The temperature after mixing may be seen in column 2 to range from 28

TABLE 8

EFFECT OF MIXING TIME AND TEMPERATURE ON RHEOLOGICAL BEHAVIOR OF FRESH CEMENT PASTES COOLED TO LESS THAN 57 C

		1st Cycle		2nd Cycle		
Mixing Time (min)	Final Temp. (°C)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
(1)	(2)	(3)	(4)	(5)	(6)	(7)
1	28	210	0.57	210	0.56	-0.018
2	32	200	0.53	190	0.52	-0.019
3	35	250	0.62	250	0.62	0
4	37	280	0.68	280	0.68	0
5	39	310	0.72	300	0.71	-0.014
8	47	310	0.73	300	0.72	-0.014
10	49	350	0.80	345	0.80	0
15	57	680	1.47	665	1.43	-0.028

TABLE 9

EFFECT OF MIXING TIME AND TEMPERATURE ON RHEOLOGICAL BEHAVIOR OF FRESH CEMENT PASTES COOLED TO LESS THAN 48 C

		1st Cycle		2nd Cycle		
Mixing Time (min)	Final Temp. (°C)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
(1)	(2)	(3)	(4)	(5)	(6)	(7)
1	24	230	0.62	230	0.60	-0.033
2	27	280	0.66	280	0.67	+0.015
3	24	320	0.75	325	0.76	+0.013
4	22	315	0.74	320	0.76	+0.027
5	23	400	0.92	410	0.94	+0.022
8	34	535	1.18	520	1.19	+0.008
10	40	595	1.41	540	1.40	+0.007
15	48	970	2.14	720	2.10	-0.019

to 57 C for mixing times (col. 1) of 1 to 15 min. In the second set of experiments (Table 9) ice was added and the temperatures ranged from 22 to 48 C (col. 2) for similar mixing times. The total time between the placing of the cements in contact with water and the making of measurements was 15 min. After mixing the cements with water, the samples were allowed to stand in the constant temperature bath at 25 C.

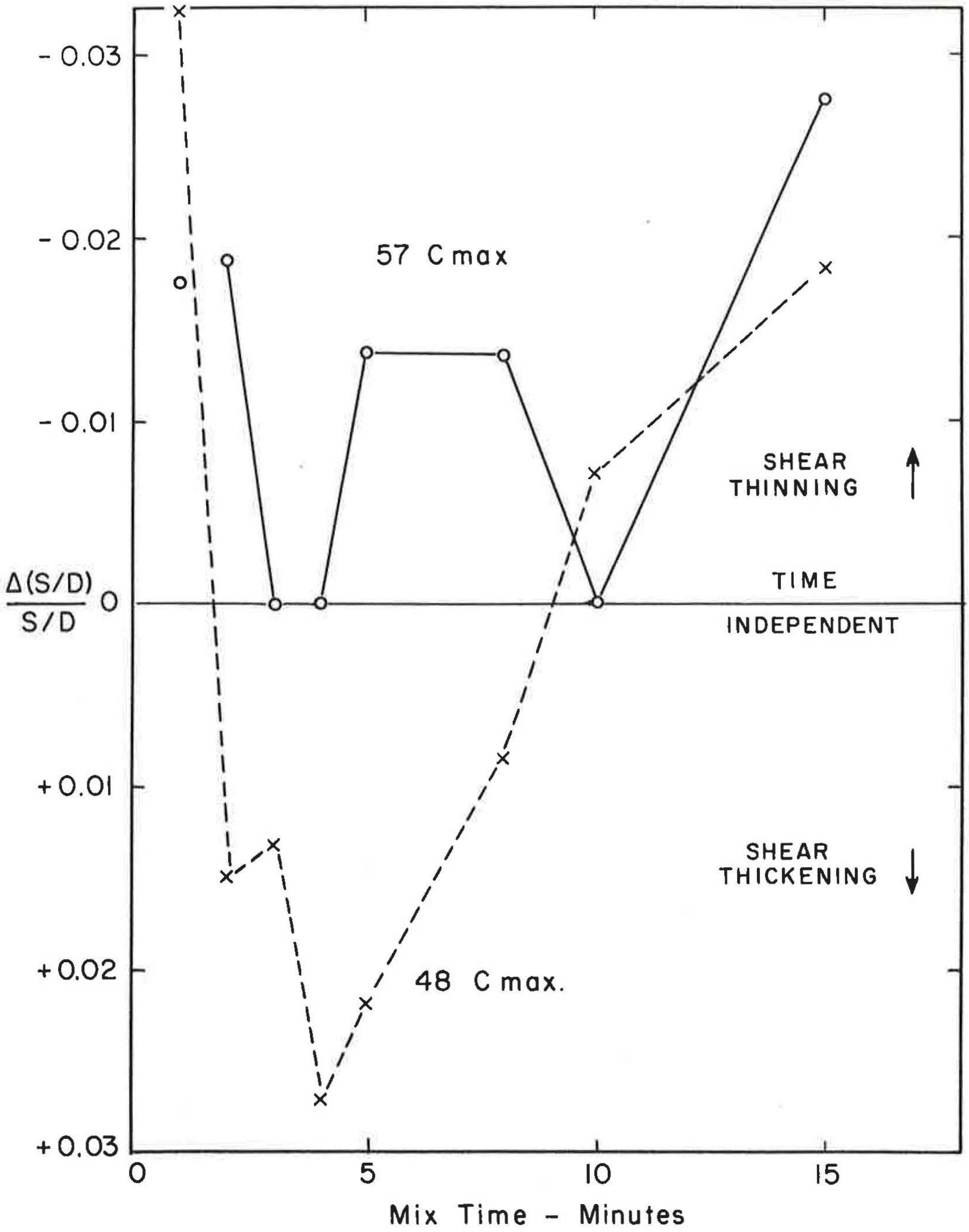

Figure 6. Effects of temperature and mixing times on flow properties of cement pastes.

In the case of the set B experiments (Table 9) for mixing periods of 1 to 5 min, the temperatures were 25 ± 3 C (col. 2). It is therefore possible to assume that for these experiments the temperature was essentially constant, and the effects of mixing time alone can be observed. For the first cycle (cols. 3 and 4) the yield values increase from 230 to 400 dynes per sq cm and the apparent viscosities increase from 0.62 to 0.92 dynes-sec per sq cm. Except for the case of the 1-min mixing time experiment, all the others exhibited a slight shear thickening behavior. Figure 6 shows that the Δ(S/D)/(S/D) values increase with mixing time up to 4 min.

For mixing times of from 8 to 15 min with temperatures below 48 C (Table 9), the yield values and apparent viscosities in the first cycle increased from 535 to 970 dynes per sq cm and 1.18 to 2.14 dynes-sec per sq cm, respectively. For the range of mixing times 4 to 15 min (Fig. 6) the Δ(S/D)/(S/D) values decreased from +0.027 to -0.019. In other words, with mixing time the system changed from a shear thickening to a shear thinning behavior.

In the experiments conducted without the addition of ice (Table 8) the pastes exhibited

TABLE 10

EFFECT OF BLENDING IN $CaSO_4$ ON RHEOLOGICAL PROPERTIES OF A CLINKER

Type of $CaSO_4$	1st Cycle: Yield Value f (dynes/sq cm)	1st Cycle: Apparent Viscosity S/D (dynes-sec/sq cm)	2nd Cycle: Yield Value f (dynes/sq cm)	2nd Cycle: Apparent Viscosity S/D (dynes-sec/sq cm)	Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
		Not Blended			
(1)	(2)	(3)	(4)	(5)	(6)
Gypsum	790	1.69	750	1.63	-0.036
Hemihydrate	600	1.47	540	1.47	0
Sol. anhyd.	410	1.17	390	1.11	-0.053
		Blended			
Gypsum	200	0.51	200	0.52	+0.019
Hemihydrate	270	0.70	270	0.69	-0.014
Sol. anhyd.	245	0.61	250	0.62	+0.016

TABLE 11

EFFECT OF SURFACE AREA ON RHEOLOGICAL PROPERTIES OF FRESH CEMENT PASTE

Surface Area (sq cm/g)	1st Cycle: Yield Value f (dynes/sq cm)	1st Cycle: Apparent Viscosity S/D (dynes-sec/sq cm)	2nd Cycle: Yield Value f (dynes/sq cm)	2nd Cycle: Apparent Viscosity S/D (dynes-sec/sq cm)	Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
(1)	(2)	(3)	(4)	(5)	(6)
1,100	30	0.18	25	0.14	-0.250
1,700	90	0.30	90	0.31	+0.033
1,800	150	0.43	150	0.44	+0.023
2,200	360	0.83	370	0.85	+0.024

a rise in yield values from 210 to 680 dynes per sq cm and in apparent viscosities from 0.57 to 1.47 dynes-sec per sq cm in the mixing time range 1 to 15 min. The $\Delta(S/D)/(S/D)$ values in Figure 6 demonstrate essentially a shear thinning behavior, but only above a 10-min mixing time was there an apparent increase in shear thinning behavior with mixing time. Below a 10-min mixing time no correlation between shear thinning behavior and mixing time is apparent.

During the course of the experiments it was found that there were differences in flow behavior when the procedure was varied for adding the calcium sulfates and clinker to the water. The results obtained when the solids were blended before adding them to the water were entirely different from those obtained when the calcium sulfate was added to the water before the clinker. When gypsum, hemihydrate, or soluble anhydrite were blended with clinker before addition to the water, the yield values and apparent viscosities were much lower than the values obtained when the solids were added separately (Table 10). In each case the sulfate added contained sufficient $CaSO_4$ to be 3 percent by weight of the total solids. The pastes with gypsum or soluble anhydrite, in which the components were not blended, exhibited shear thinning properties, whereas the corresponding blended pastes showed shear thickening properties (col. 6). The pastes containing hemihydrate exhibit either a time-independent or slightly shear thinning behavior.

Surface Area

The effect of the surface area of a cement on the flow properties was examined. Pastes of cement 15366 with Wagner surface areas of 1,100, 1,700, 1,800, and 2,200 sq cm per g were investigated. In these experiments a w/c ratio of 0.5 was used. After the cement was mixed with water for 3 min, it was allowed to stand at 25 C for 15 min. Table 11 shows the yield values and apparent viscosities (cols. 2 and 3) in the first cycle increase with surface area from 30 to 360 dynes per sq cm and from 0.18 to 0.83 dynes-sec per sq cm, respectively. The $\Delta(S/D)/(S/D)$ values (col. 6) indicate only that with an increase in surface area there is a change from shear thinning to a shear thickening behavior.

Various Cements and Components

This series of experiments examined (a) Type I cement 15366, (b) a high alkali

TABLE 12

RHEOLOGICAL PROPERTIES OF VARIOUS CEMENTS AND CALCIUM SILICATES

Sample	Number	1st Cycle Yield Value f (dynes/sq cm)	1st Cycle Apparent Viscosity S/D (dynes-sec/sq cm)	2nd Cycle Yield Value f (dynes/sq cm)	2nd Cycle Apparent Viscosity S/D (dynes-sec/sq cm)	Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
(1)	(2)	(3)	(4)	(5)	(6)	(7)
-	15366	400	0.94	400	0.95	+0.011
High alkali	15698	320	0.94	320	0.86	-0.890
Clinker	15367[a]	-	-	-	-	-
Med. C_3A	15622A	65	0.27	60	0.26	-0.038
High C_3S	15497B	435	1.01	430	1.01	0
Low C_3A	15669	90	0.37	90	0.35	-0.056
Zero C_3A	19648	90	0.37	90	0.36	-0.027
C_3S	-	40	0.22	35	0.20	-0.095
C_2S	-	80	0.34	80	0.31	-0.092

[a] Too stiff to measure.

cement 15698, (c) a clinker 15367, (d) a medium C_3A cement 15622A, (e) a high C_3S cement 15497B, (f) a low C_3A cement 15669, (g) a cement with no C_3A (19648), (h) C_3S, (i) C_2S, and (j) mixtures of C_3A, calcium sulfates, and quartz. Table 1 gives the computed compound compositions of the cements. The solids were shaken with the water for 5 min and then allowed to stand for 15 min at 25 C before the viscometer measurements were made. The concentrations of solids correspond to a water to solid ratio of 0.5.

The results of the experiments with the cements, C_3S and C_2S are given in Table 12. The $\Delta(S/D)/(S/D)$ values (col. 7) show a slight shear thickening behavior for pastes of cement 15366. The other cement pastes exhibited shear thinning properties of a time-independent behavior. In the case of the clinker paste, the system was too stiff to make quantitative viscometer measurements, but a shear thinning behavior was observed. The yield values in the first cycle (col. 3) for pastes of cement 15366, high alkali cement 15698 and a high C_3S (60.1 percent) cement are 400, 320, and 435 dynes per sq cm, respectively. The apparent viscosities for these cements are 0.94, 0.94 and 1.01 dynes-sec per sq cm, respectively. Pastes containing a medium C_3A cement, a low C_3A cement, a zero C_3A cement, C_3S and C_2S exhibited much lower yield values and apparent viscosities (cols. 3 and 4). The yield values and apparent viscosities for the latter pastes ranged from 40 to 90 dynes per sq cm, and from 0.22 to 0.37 dynes-sec per sq cm, respectively.

In the experiments with mixtures of C_3A, quartz, and gypsum or hemihydrate, the $CaSO_4$ contents were 3 percent and the C_3A contents varied between 0 and 5 percent of the weight of the quartz. The weight of quartz was reduced by the weight of the C_3A and calcium sulfate added in the 43 volume percent quartz pastes. The results are summarized in Table 13.

The control experiment with 2 percent C_3A showed a relatively low yield value of 100 dynes per sq cm, an apparent viscosity of 0.43 dynes-sec per sq cm in the first

TABLE 13

EFFECT OF C_3A ON RHEOLOGICAL PROPERTIES OF A QUARTZ-CALCIUM SULFATE-WATER SYSTEM

	1st Cycle		2nd Cycle		
Conc. C_3A (%)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Yield Value f (dynes/sq cm)	Apparent Viscosity S/D (dynes-sec/sq cm)	Over-All Behavior $\frac{\Delta(S/D)}{S/D}$
		Gypsum			
(1)	(2)	(3)	(4)	(5)	(6)
0	0	0.20	0	0.19	-0.051
1	640	1.55	660	1.59	+0.026
3	810	1.91	830	1.91	0
5	1,380	3.26	1,290	3.00	-0.083
		Hemihydrate			
0	160	0.69	180	0.76	+0.097
1	1,390	3.13	1,330	3.16	+0.010
2	1,240	2.92	1,230	2.95	+0.010
3	1,320	3.01	1,300	3.04	+0.010
5	1,420	3.41	1,450	3.35	-0.018
		Control			
2	100	0.43	100	0.44	+0.023

cycle (cols. 2 and 3) and a very small shear thickening behavior (col. 6). Both the yield values and apparent viscosities for the other pastes increased with an increase in C_3A content (cols. 2 and 3). The pastes with hemihydrate exhibited, in general, a very small shear thickening behavior, whereas the $\Delta(S/D)/(S/D)$ values for the pastes with gypsum demonstrated mostly a time-independent or shear thinning behavior. An increase in C_3A content would be expected to reduce the amount of plaster set. Some evidence for this behavior is found in the experiments with hemihydrate. In these experiments the yield values and apparent viscosities increase with the amount of C_3A added, and the $\Delta(S/D)/(S/D)$ values decrease from +0.097 to -0.018. No such correlation between C_3A content and $\Delta(S/D)/(S/D)$ values was found for the pastes with gypsum. Therefore, it may be concluded that C_3A decreases the shear thickening properties and increases the yield values and apparent viscosities of pastes with dehydrated gypsum.

ANALYSIS

Normally, cement pastes stiffen gradually as the C_3S hydrates. The formation of sulfoaluminate accelerates the stiffening. When the hydration of C_3A and C_4AF occurs without the inhibiting action of the calcium sulfate, an abnormal stiffening referred to as quick set is developed. The calcium sulfates inhibit the hydration of the aluminates, but when dehydrated gypsum is present an abnormal stiffening may be produced by the deposition of gypsum crystals in a plaster set. With these concepts, it is possible to explain most of the results of this investigation.

The stiffening of quartz pastes, as shown by the yield values and apparent viscosities (Table 4), is produced by hemihydrate and soluble anhydrite. Insoluble anhydrite and gypsum produce much smaller effects. Therefore, as a result of the greater solubilities of soluble anhydrite and hemihydrate, a larger deposition of gypsum crystals occurs with these two sulfates. When more than 50 percent of the $CaSO_4$ in a gypsum-hemihydrate mixture is hemihydrate the pastes begin to exhibit much higher yield values and apparent viscosities (Fig. 3).

Without added calcium sulfate, a clinker paste stiffens immediately. When gypsum is present in a clinker paste, the yield values and apparent viscosities (Tables 5 and 6) are relatively low. Pastes containing hemihydrate and soluble anhydrite exhibit yield values and apparent viscosities that are much larger. The addition of insoluble anhydrite to clinker pastes produces yield values and apparent viscosities that are larger than those exhibited by any of the pastes with the other calcium sulfates. When hemihydrate-gypsum mixtures are added to clinker pastes the yield values and plastic viscosities (Table 5 and Fig. 4) decrease at first with an increase in hemihydrate content. At a 60 percent hemihydrate content the values begin to increase rapidly. These results may be explained by the concepts previously presented. Gypsum alone can provide clinker pastes with sufficient sulfate to inhibit to a large extent the rapid hydration of C_3A and C_4AF. It should be recalled that the gypsum contained about 8 percent hemihydrate. As the hemihydrate content in the mixture is increased to 60 percent (Fig. 4), the hydration of C_3A and C_4AF is further inhibited. When there is more than 60 percent hemihydrate in the mixture, the increase in yield values and apparent viscosities may be attributed to plaster set. Pastes with hemihydrate and soluble anhydrite additions show larger yield values and apparent viscosities than those with gypsum, because the higher solubilities of the dehydrated forms cause plaster set. Insoluble anhydrite, on the other hand, does not dissolve rapidly enough to inhibit completely the hydration of the C_3A and C_4AF.

The experiments with LTS No. 18 cement heated to 120 C clearly show the increases in yield values and apparent viscosities (Table 7 and Fig. 5) caused by dehydrated gypsum.

It was not possible to demonstrate a clear relationship between the shear thickening properties of fresh cement pastes and plaster set. Hemihydrate did produce a change from shear thinning to shear thickening properties in quartz pastes when the hemihydrate content in the gypsum-hemihydrate mixtures exceeded 50 percent (Table 3 and Fig. 3). In addition, hemihydrate or soluble anhydrite alone produced shear thickening flow behaviors in quartz pastes (Table 4). There is no consistent trend shown between

$\Delta(S/D)/(S/D)$ values and sulfate contents. The values appear first to rise and then to decrease with increasing sulfate contents. Insoluble anhydrite and gypsum produced time-independent or slightly shear thinning properties in quartz suspensions.

Hemihydrate or soluble anhydrite caused only some indication of a shear thickening behavior in clinker pastes (Tables 5 and 6). Pastes with gypsum added exhibited time-independent or a slight shear thickening behavior. The shear thinning behavior of a clinker paste to which insoluble anhydrite was added may be attributed to hydrated calcium aluminates because the formation of these hydrates is not very well inhibited by the slowly dissolving insoluble anhydrite.

Pastes of LTS No. 18 cement heated at 120 C showed (Table 7 and Fig. 5) a shear thickening behavior for a longer period of hydration time than that exhibited by pastes of the unheated cement. This effect may be attributed to the large amount of plaster set obtained with the cements containing completely dehydrated gypsum.

The shear thinning behavior of cement pastes seems to increase with mixing time and the temperature after mixing (Tables 8 and 9). Although 1-min mixing time is necessary to produce a shear thickening behavior, a mixing period longer than 4 min reduces the shear thickening properties. These effects may be attributed perhaps to the influences of temperature and mixing time on the rates of hydration and on the breaking up of the plaster set.

It is necessary to blend the clinker and gypsum, or soluble anhydrite, before adding them to water to produce even a small amount of shear thickening behavior (Table 10). Apparently the sulfate ions are not made readily available unless the sulfates are blended with the clinker before addition to the water.

The surface area of the cement influences the flow behavior. Shear thickening properties were only found with the higher surface area cement samples (Table 11). Nevertheless, no correlation between the degree of shear thickening behavior and surface area was observed. The influence of surface area may be attributed to the changes it produces in rates of hydration and solid-solid and solid-water interactions.

Only cement 15366 with no special characteristics exhibited a shear thickening behavior (Table 12). Shear thinning behaviors were demonstrated by the other cements, C_3S and C_2S. C_3A reduced (Table 13) the shear thickening properties of a hemihydrate-quartz suspension. This may be attributed to the removal of sulfate from solution by the C_3A, which reduced the degree of plaster set. The hydration of C_3A to C_4AH_{19} leads to an increase in the yield values, apparent viscosities, and shear thinning properties of quartz suspensions containing C_3A. The larger yield values and apparent viscosities produced by C_3A in hemihydrate-quartz suspensions than in gypsum-quartz suspensions is probably due to the presence of plaster set and a larger amount of sulfoaluminate crystals. The shear thinning behaviors of the cement pastes increased with the C_3A and C_3S contents of the cements (Table 12).

Whereas for quartz-calcium sulfate suspensions the relation between plaster set and shear thickening behavior was clearly demonstrated, the relation for the clinker-calcium sulfate system is not nearly so clear. Nevertheless, there is strong indication in the data presented that such a relation probably does exist. However, the clinker-calcium sulfate system is so complex and there are so many factors that have bearing on the problem, that only further research could lead to a full understanding of the shear thickening behavior. One factor, for example, on which no information exists at present is the influence of the sulfoaluminate on the flow properties of fresh pastes; it is possible that sulfoaluminates play an important role in early abnormal stiffening.

In future rheology studies both suspensions of components and mixtures of the components of cement should be examined. Such studies will lead to a better understanding of the factors that influence the flow behaviors of fresh cement pastes.

Mixtures of gypsum and mineral anhydrite have been used to reduce abnormal stiffening (2). In this investigation it was demonstrated that in the range 40 to 60 percent hemihydrate content in hemihydrate-gypsum additions to clinker pastes, the lowest yield values and apparent viscosities were found (Table 5 and Fig. 4). Therefore, it is possible to conclude that when mixtures of gypsum and the mineral anhydrite are ground with clinker, hemihydrate is produced by dehydration of some of the gypsum. When the proper mixture of the two sulfates is added to clinker there is sufficient soluble hemihydrate to react immediately with the C_3A to inhibit quick set, but not sufficient to produce a plaster set. The mineral anhydrite and the remaining gypsum in mixtures could then react slowly with the C_3A.

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Shrinkage of Concrete—Comparison of Laboratory and Field Performance

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In general, portland cement concrete, on drying, does not develop an amount of shrinkage detrimental to adequate service; however, there are cases where serviceability is known to have suffered due to an abnormal amount of shrinkage of the hardened concrete. Such distress has occurred in both structures and pavements. One reason for the poor performance was that the acceptability of the work in progress was based largely on strength tests that bore little relationship to shrinkage.

In this paper, factors producing shrinkage of laboratory specimens are reviewed from the literature and supplemented by original test data. Of the many factors contributing to shrinkage, those that are controllable on the work under typical specifications are of less importance than the characteristics of the constituent materials for which the usual specification requirements provide inadequate protection. A combination of unfavorable conditions and materials can increase shrinkage fourfold, an amount conducive to poor performance.

The results of laboratory specimens dried under standard conditions are compared to those of field exposure of full-size or near full-size structures and pavements. It is shown that by proper design of laboratory tests, the performance of job concrete can be predicted with assurance. The need of specification requirements against high shrinkage produced by cements and aggregates and more restrictive requirements for admixtures is demonstrated.

• CONSTANCY of volume of concrete after it has hardened has long been recognized as essential to satisfactory performance. Early studies, however, have been concerned mainly with provisions to avoid long-time expansion in the presence of moisture rather than shrinkage on drying. Requirements have been in effect to avoid excessive expansion due to excess amounts of free lime, magnesia, and SO_3 in portland cement, and to chemical reactions between alkalies and certain constituents of aggregates. The presence of sulfates in the surrounding environment has also been recognized as a potential cause of detrimental expansion, as has the effect of frequent cycles of freezing and thawing.

On the other hand, the effect of shrinkage due to loss of moisture on drying has not received the amount of attention that the subject warrants. One reason for this situation is due to the emphasis placed on strength as a measure of the desirable properties of concrete. Too often the fact has been overlooked by designers and construction engineers that shrinkage bears little relationship to strength. This condition has prevailed despite a statement in the ACI Manual of Concrete Inspection (1) that a principal requirement of hardened concrete is that it should not shrink excessively on drying.

Because in most cases there is little easily recognized adverse effect of shrinkage, many engineers and architects have been prone to disregard it as a factor requiring special attention. Nevertheless, evidence in U. S. literature of unsatisfactory per-

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formance dates back as early as 1924 (2). The detrimental effects of shrinkage of concrete in pavements was emphasized by Hveem (3) in 1951. Tremper gave illustrations of unsatisfactory performance in pavements due to excessive shrinkage in a discussion of a paper by Jackson (4) in 1950 and in structures in the author's closure of a paper (5) in 1959.

The subject of shrinkage is now receiving greater attention, as evidenced by two recent articles in Engineering News-Record (6, 7) and by a British report (8) published in 1961.

Laboratory studies of shrinkage are fairly numerous and they point out the fact that shrinkage is influenced by many factors. What is lacking is a thorough investigation of the significance of laboratory test results with respect to performance of concrete in service. There is also a need of perspective as to which factors have greater adverse effects and which are more readily susceptible to control in a practical manner. This report attempts to answer such questions.

In accordance with the growing custom in the United States, the term "shrinkage" as used in this report refers to the shortening of hardened concrete, as measured on one axis, which results mainly from loss of moisture due to drying. It is recognized that the measured shortening may also partly result from carbonation, but in the early stages of drying the effects of carbonation are believed to be relatively minor. Carbonation shrinkage, as such, is only incidentally discussed in this report.

The studies presented here of the field performance of concrete as affected by drying relate generally to exterior exposure and specifically to highway pavements and building structures. A distinction between pavements and buildings is made because certain members in heated or air-conditioned buildings may be expected to reach a more advanced state of dryness than concrete exposed entirely to outdoor weather does. Further, portions of buildings are subjected to an intermediate degree of exposure. Examples are exterior walls, floor slabs on grade, and the lower portions of interior walls and columns.

The first part of this report deals with the shrinkage of laboratory-made specimens dried under controlled and uniform conditions. The second part deals with the performance of pavements and structures subjected to drying under exterior exposure, and with the relationship between laboratory and field performance. Methods of making laboratory tests for shrinkage to obtain meaningful results in a minimum of elapsed time are discussed.

Original data in this report are from tests made in the laboratory of the California Division of Highways referred to as "California." Unless otherwise noted, mortar shrinkage tests were made on 1:2 graded Ottawa sand mortar having a flow of 100 to 115 percent. Specimens were 1- by 1- by $11\frac{1}{4}$-in. prisms with gage studs giving a gage length of 10 in. Four specimens were made from each batch of mortar. They were removed from the molds at 24 hr, placed in water at 73.4 ± 3F to the age of 72 hr, and then measured for length. They were placed on racks in a drying room maintained at 73.4 ± 2F, with a relative humidity of 50 ± 4 percent and with air movement. This resulted in evaporation of water from an atmometer at 3 ± 0.5 ml per hr. At an age of 7 days, they were again measured for length. The average decrease in length during drying expressed as a percentage of 10 in. is reported as "shrinkage." Further details of the method are given in Test Method No. Calif. 527, a publication of the California Division of Highways.

Unless otherwise noted, concrete shrinkage tests were made of 7-sack, $3\frac{1}{2}$-in. slump concrete, using a blend of equal parts of Type II, low-alkali cements from five California mills, and washed aggregates of uncrushed sand and gravel from the American River near Sacramento, after sieving and recombining to a predetermined grading of 1-in. maximum size. Specimens were 3- by 3- by $11\frac{1}{4}$-in. prisms with gage studs giving a gage length of 10 in. Three specimens were made from each batch of concrete. They were removed from the molds at 24 hr and stored in a fog room at 73.4 ± 3F to the age of 7 days when they were measured for length. They were then placed on racks in the drying room described for mortar specimens, to an age of 21 days (14 days of drying), when they were again measured for length. The decrease in length during drying, expressed as a percentage of 10 in. of at least 7 specimens of 9, is reported as "shrinkage." Further details of the method (specifically written for testing admixtures) are given in Test Method No. Calif. 530, a publication of the California Division of Highways.

LABORATORY PERFORMANCE

Concrete Composition

Tests by the U. S. Bureau of Reclamation (9) show that shrinkage is related primarily to the unit water content of concrete. Variations in slump and in the grading of aggregates affect shrinkage by virtue of their effect on unit water content. Walker and Bloem (10) found that the water requirement increased with decreasing maximum size of aggregate and that shrinkage increased, a finding that was confirmed by Tremper (10) in his discussion of that paper.

Tests by the U. S. Bureau of Reclamation (9) show that an increase in entrained air (up to 5 percent) has virtually no effect on shrinkage if the slump of the concrete is held constant. Keene (11) also found this to be true, as did California.

Davis (12) reported that some data show marked increase in shrinkage in richer mixes, whereas other data do not show much change. Carlson (13) found that, when a mixed gravel aggregate was used, the cement factor had little effect on shrinkage. On the other hand, he found that rich concretes containing dolomite and granite aggregates (and by inference, possibly all aggregates of high rigidity) shrink more than the corresponding concretes of lower cement factor do.

Lyse (14) reported that shrinkage is proportional to the volume of paste:

$$S = bp \qquad (1)$$

in which

b = a coefficient; and
p = absolute volume of water plus cement.

Further, he reported that the quality of paste has little, if any, effect on shrinkage per unit of paste. Pickett (15) found that shrinkage is related to a function of the absolute volume of aggregate:

$$S = b\,(1 - g)^{1.7} \qquad (2)$$

in which

b = a coefficient; and
g = absolute volume of aggregate.

In the absence of air (or if air is calculated as part of the paste), $p = 1 - g$. Whereas Eq. 1 states that shrinkage is directly proportional to the volume of paste, Eq. 2 states that shrinkage is proportional to the 1.7th power of the paste volume.

Lyse apparently took no account of air content, his values for paste evidently being the sums of the absolute volumes of cement and water. Pickett did not report air contents, but he concluded that high air contents due to poor compaction were responsible for those observed shrinkages that were greater than predicted by his equation.

Pickett worked entirely with mortar. Lyse worked with concrete of unreported maximum size of aggregate, but the size was not large because his test specimens were 3- by 6-in. cylinders.

Tests made by California for effect of cement factor (Table 1) were made with concrete containing aggregate of 1-in. maximum size. The percentage of fine aggregate was reduced progressively with increasing cement factor in accordance with ACI Standard 613 (16).

The data show that the volume of paste increased and the volume of aggregate decreased with increasing cement factor, and that shrinkage did not change (at least not in the direction indicated by the equations of Lyse and Pickett). The volume of water plus air varies but little, and the volume of water alone is nearly as constant. The coefficient of variation in shrinkage from the mean value, due to changes in cement factor, is 6.7 percent. The data, therefore, appear to indicate that with the aggregate

TABLE 1

EFFECT OF CEMENT FACTOR ON SHRINKAGE OF CONCRETE[1]

Cement Factor (sacks/cu yd)	Concrete Composition by Absolute Volume[2]						Water-Cement Ratio by Wt.	Slump (in.)	Shrinkage[3] (%)
	Cement	Water	Air	Total (paste)	Aggregate	Water + Air			
4.99	0.089	0.202	0.017	0.308	0.692	0.219	0.72	3.3	0.0330
5.99	0.107	0.207	0.016	0.330	0.670	0.223	0.62	3.6	0.0330
6.98	0.124	0.210	0.014	0.348	0.652	0.224	0.54	3.8	0.0289
8.02	0.143	0.207	0.015	0.365	0.635	0.223	0.46	3.8	0.0300
Summary of variances:									
Mean of values		0.2065				0.222			0.0312
Std. dev.		0.003				0.002			0.0021
Coeff. of variation (%)		1.5				0.9			6.7

[1] American River sand and gravel graded to 1-in. maximum.

[2] Average of 3 batches.

[3] Average of nine 3- by 3- by 10-in. prisms cured wet for 7 days, then dried for 14 days.

used, in the range of practical concrete mixes, the cement factor has little effect on shrinkage. Because the unit water content is nearly constant, the data are in agreement with the statement made in the Concrete Manual of the U. S. Bureau of Reclamation that shrinkage is governed mainly by unit water content.

California made tests of graded Ottawa sand mortar in the proportions of 1:3, 1:2½, 1:2, and 1:1½ (Table 2). Corresponding cement factors were 8.0, 9.3, 11.2, and 13.5 sacks per cubic yard. The data show that the trends are similar to those found for concrete mixtures. The volume of the paste increased and the volume of the sand decreased with increasing cement factor. Although the shrinkage of the 1:1½ mortar is slightly higher, that of the remaining mortars is essentially constant.

Coefficients of variation from the mean of the four mixtures are as follows:

Variable	Percent
Unit volume:	
Water	2.3
Water + air	1.7
Shrinkage:	
Dried 4 days	5.9
Dried 7 days	3.2

Although an analysis of variance shows the effect of the cement factor to be significant with respect to shrinkage, this finding is due to the relatively high shrinkage found in the 1:1½ mix. It seems more logical to conclude that shrinkage is related to the unit volume of water or water plus air within customary mortar mixtures, and that because these values are constant, the shrinkage is also constant. This conclusion agrees with that derived from the California tests of concrete.

Cement

Carlson (13) found difficulty in measuring the shrinkage of neat cement paste because cracking developed and obscured the over-all change in length. He estimated that cement paste, if unrestrained, would shrink from 5 to 15 times as much as does concrete.

TABLE 2

EFFECT OF CEMENT FACTOR AND DURATION OF PRELIMINARY WET CURING ON SHRINKAGE OF MORTAR[1]

Cement-Sand Ratio by Wt.	Mortar Composition by Absolute Volume[2]						Shrinkage[3] (%)							
							4-Day Drying				7-Day Drying			
	Cement	Water	Air	Total (paste)	Sand	Water + Air	2-Day Wet Curing	3-Day Wet Curing	7-Day Wet Curing	Average	2-Day Wet Curing	3-Day Wet Curing	7-Day Wet Curing	Average
1:3	0.155	0.242	0.057	0.453	0.547	0.299	0.0421	0.0410	0.0416	0.0416	0.0545	0.0544	0.0550	0.0546
1:2½	0.177	0.246	0.051	0.474	0.526	0.297	0.0420	0.0391	0.0388	0.0400	0.0547	0.0519	0.0528	0.0535
1:2	0.211	0.252	0.037	0.500	0.500	0.289	0.0428	0.0411	0.0407	0.0415	0.0560	0.0536	0.0533	0.0543
1:1½	0.256	0.255	0.033	0.544	0.456	0.288	0.0469	0.0458	0.0442	0.0458	0.0589	0.0572	0.0564	0.0575
Avg.							0.0435	0.0418	0.0413	0.0423	0.0560	0.0542	0.0544	0.0549

[1] Type II, low-alkali cement; graded Ottawa sand mortar, flow 100 to 115 percent.
[2] Average of 3 batches.
[3] Average of 12 specimens from 3 batches.

Carlson found that among the potential compounds in portland cement, their relative contribution to shrinkage in increasing order is C_3S, C_2S, and C_3A.

Carlson (13) found that an increase in specific surface of the cement increases shrinkage, but that fineness has less effect on cements very low in C_3A.

Swayze (19) reported shrinkage tests of concrete specimens containing 40 cements. After drying for 25 days, the shrinkage of one cement exceeded that of another by 68 percent. The average shrinkage of the group of 10 cements having the highest shrinkage exceeded that of the group of 10 cements having the lowest shrinkage by 25 percent.

Lyse (14) found that Type III cements give higher shrinkage than Type I cements in lean concrete (W/C = 1.00), but that in a rich concrete (W/C = 0.50), the difference in shrinkage is small.

California found that the average shrinkage of the Type I cements investigated was 119 percent of that of Type II cement, the determination being made on 1- by 1- by 10-in. specimens of 1:2 graded Ottawa sand mortar that were dried for 4 days following 3 days of water curing. Among Type III cements from 7 mills, similarly tested, the range in shrinkage was from 0.045 to 0.083 percent, and the average value was 0.065 percent. Only one Type III cement gave a shrinkage lower than 0.048 percent, which is the maximum under California Division of Highways specifications for Type II, low-alkali cement.

Lerch (17) demonstrated that the proportion of gypsum in the cement has a major effect on shrinkage and that when the SO_3 is in optimum amount for a particular cement, the shrinkage is the lowest.

A report by the Working Committee on SO_3 Content of ASTM Committee C-1 (18) shows that of 8 cements, each containing SO_3 at approximate optimum, the shrinkage of one exceeded that of another by 67 percent. Higher shrinkage was found in cements of higher potential C_3A and alkalies.

Haskell (20) has shown by statistical analysis that the optimum percentage of SO_3 is closely related linearly to the specific surface and the percentages of alkalies and tricalcium aluminate.

Pickett (21) concluded that the percentage of SO_3 for minimum shrinkage in concrete may be greater than that in mortar. California found that for a Type II, low-alkali cement to which additions of pulverized gypsum were made, the percentage of SO_3 for minimum shrinkage of 6-sack concrete was about 0.5 percent greater than for minimum shrinkage of 1:2 graded Ottawa sand mortar (Table 3).

Pickett (22) found that under the conditions of test, the addition of alkali hydroxide to cement decreased the rate of shrinkage of concrete. He concluded, however, that because many factors affect the rate of shrinkage, and alkalies have many other effects that may affect shrinkage indirectly, one should expect many real and apparent contradictions to his finding that an increase in alkalies retards shrinkage.

Pickett (22) also concluded that the addition of gypsum to the optimum reduces shrinkage by reducing the specific surface of the gel that is formed. He also found that cements high in alkali have a lower coefficient of shrinkage diffusivity, and that the lower coefficient produces higher shrinkage stresses and, hence, a greater tendency to crack.

The effect on shrinkage of processing additions used in the manufacture of portland cement is controlled under ASTM Designation C 465-62 T by a requirement that the shrinkage (expressed as a percentage) of mortar made with the cement containing the addition shall not be more than 0.020 greater than that developed by the cement without the addition after drying for 7 days, 28 days, and 3 months. Length change measurements are made on 2- by 2- by 10-in. specimens of 1:2 concrete sand mortar. Such specimens normally shrink about 0.04 percent in 7 days; therefore, the permissible increase in shrinkage amounts to about 50 percent at this test age.

California has determined the effect of the processing addition TDA, used as an admixture, on the shrinkage of a Type II, low-alkali cement as influenced by its content of SO_3. The cement as received contained 1.0 percent SO_3. Pulverized gypsum was added in increments to increase the SO_3 contents progressively by 0.5 percent up to 3.0 percent. Test specimens were 1- by 1- by 10-in. prisms of 1:2 graded Ottawa sand mortar which were cured wet for 3 days and then dried for 4 and 11 days. The test results are shown in Figure 1. The increase in shrinkage produced by TDA was considerably less when the SO_3 content of the cement exceeded optimum.

TABLE 3

EFFECT OF SO_3 CONTENT ON SHRINKAGE OF MORTAR AND CONCRETE[1]

SO_3 in Cement (%)	Shrinkage (%)	
	Mortar	Concrete
1.0	0.071	0.044
1.5	0.048	0.041
2.0	0.039	0.036
2.5	0.040	0.031
3.0	0.051	0.031
3.5	0.060	
4.0		0.045
4.5		0.048
Est. optimum	2.2	2.7

[1] Pulverized gypsum added in progressive amounts to Type II, low-alkali cement containing 1.0 percent SO_3. Mortar tests were 1- by 1- by 10-in. prisms of 1:2 graded Ottawa sand mortar cured wet for 3 days, then dried for 4 days. Concrete tests were 3- by 3- by 10-in. prisms of 6-sack concrete, 1-in. maximum size aggregate, 3- to 4-in. slump, cured moist 7 days, then dried for 28 days.

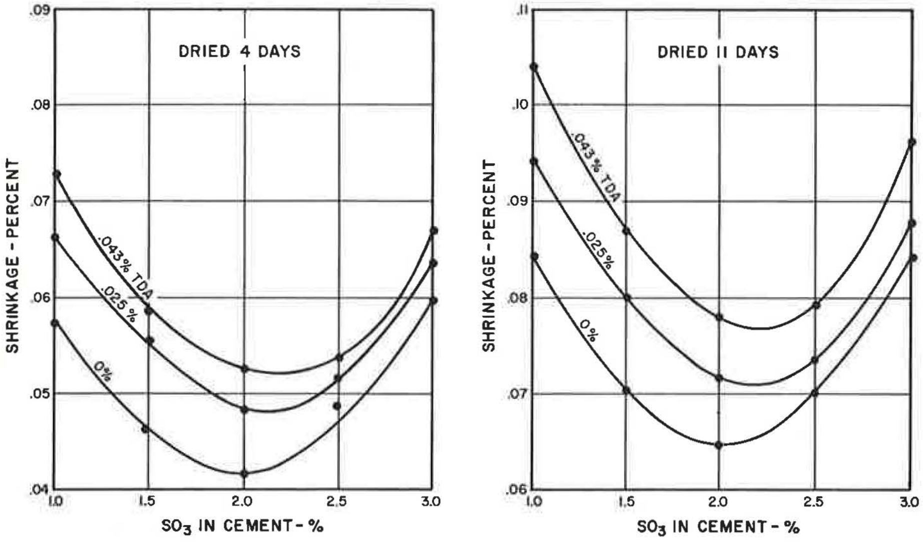

Figure 1. Influence of SO_3 in cement on shrinkage effect of TDA.

An analysis of variance applied to the data of Figure 1 indicates that a reaction occurred between the TDA and the SO_3. In a later discussion of admixtures, it is shown that the effect of variations in the amount of triethanolamine (a constituent of TDA) on the shrinkage of concrete is not proportional to the effect on mortar. It is shown that a similar situation holds with calcium chloride, which is known to react chemically with constituents of cement. These results indicate that tests for the effect of processing additions that react with cement when made on mortar, as provided in ASTM Designation C 465-62 T, are likely to be misleading as a measure of the shrinkage produced in concrete.

Aggregates

Carlson (13) studied the effect of different aggregate types on their contribution to shrinkage of concrete. He found that sandstone and slate produce high shrinkage. Among the pure mineral aggregates that were tested, hornblende and pyroxene produced the highest shrinkage. Quartz, feldspar, dolomite, and calcite produced low shrinkage.

Carlson concluded that the relative compressibility of aggregate particles appears to be the most important factor causing different aggregates to produce concrete of different shrinkage, although in a previous paper (24) he discounted the effect of aggregate compressibility. He believed that surface characteristics of aggregates and their ability to entrain air voids at the surface of the particles were of importance. He concluded that the particular properties of aggregates conducive to tight bond with cement paste remain to be determined.

Carlson (13) also found that among four aggregates studied there was a close correlation between absorption of the aggregate and shrinkage of the concrete.

Hveem and Tremper (25) found in tests of sands that higher absorption produced higher shrinkage of mortar, and that the correlation was exceptionally high. Exceptions consisted of expanded shale aggregates and sands consisting mainly of vesicular basalt.

Hveem and Tremper (25) found that the content of clay-like particles in sands, as determined by the sand equivalent test, is an important factor contributing to shrinkage. Mortar containing sand of 70 sand equivalent was found to shrink twice as much as one made with sand of 95 sand equivalent. When the effects of sand equivalent and absorption were considered together, the correlation was found to be significant at the 0.1 percent level. They also found that the presence of clay-like particles in coarse aggregate increases the drying shrinkage of concrete. The contribution of clay-like particles to shrinkage was found to be in excess of that expected purely by an increase in unit water content.

California has determined the shrinkage of concrete containing a variety of sand and gravel aggregates produced in the State. Table 4 gives the tested aggregates in ascending order of shrinkage. The tests were made on aggregates of the same grading that were comparably free from clay-like particles. Differences in shrinkage cannot be accounted for solely by differences in absorption or percentage of wear in the Los Angeles abrasion machine. Expanded shale aggregates (produced in California), not included in Table 4, produced relatively low shrinkage. It thus appears that there are properties of aggregates that are not measured in the commonly made tests but which have a large effect on concrete shrinkage.

Roper (26) investigated the volume changes of mortars and concretes containing certain South African aggregates which had a poor service record. He found that the aggregates that produced high shrinkage in mortar or concrete were themselves dimensionally unstable when dried and subsequently wetted. He noted that many rocks being used as concrete aggregate in the United States evidence dimensional instability; a few, of serious degree.

Admixtures

Many powdered admixtures require an increase in the unit water content of concrete and for this reason may be expected to increase shrinkage (23). As noted earlier, the air entrained by air-entraining admixtures has virtually no effect on shrinkage.

The effect on shrinkage of chemical admixtures is varied and cannot be correlated with a possible reduction in unit water content, which is often substantial in amount. Many, but not all, chemical admixtures of the set-retarding type reduce shrinkage or increase it only slightly (not more than about 10 to 20 percent) (Table 5). On the other hand, some set-retarding admixtures and most lignin-base admixtures that have been treated to destroy set-retarding properties, when used at the manufacturer's recommended dosage, produce substantial increases in shrinkage, up to 60 percent when measured in concrete specimens dried for 14 days. The relative effect after longer periods of drying is discussed later.

Two compounds frequently used to destroy the set-retarding effect of chemical admixtures and to increase the rate of strength development are calcium chloride and triethanolamine. California test results for the effect of these admixtures are given in Tables 6 and 7, and Figures 2 and 3. The effect on shrinkage is disproportionately large with small amounts of these admixtures, thus demonstrating that their inclusion in other types of admixtures can seriously increase drying shrinkage even though used in relatively small amounts as a percentage of the cement.

Figures 4 and 5 show that the effect of calcium chloride and triethanolamine on the shrinkage of concrete is not directly proportional to the effect on the shrinkage of mortar.

There is evidence that many chemical admixtures react with the cement, thus forming compounds that are not otherwise present. This is a probable reason that many of them increase shrinkage while also reducing the unit water content.

TABLE 4

RELATIVE SHRINKAGE OF CONCRETE MADE WITH DIFFERENT AGGREGATES[1]

Aggregate (Ref. No.)	Drying Shrinkage[2] (% of control)
1	70
2	76
3	79
4	81
5	83
6	86
7	89
8	89
9	92
10	94
11	95
12	97
13	97
14	97
15	97
16	97
17	100
18	106
19	107
20	110
21	110
22	110
23	112
24	120
25	124
26	129
27	130
28	133
29	145
30	153

[1] Five-sack concrete, 2-in. slump, 1½-in. maximum size aggregate. Specimens were 4- by 5- by 18-in. prisms, moist cured for 7 days, then dried for 28 days. All aggregates were washed, then sieved and recombined to same grading.

[2] Average of 2 to 5 specimens expressed as percentage of control concrete containing aggregate from American River.

Duration of Moist Curing

Carlson (13) reported that the duration of preliminary moist curing does not have much effect on shrinkage. Lyse (14) found that increasing periods of moist curing up to 7 days increased shrinkage, but that further curing up to 28 days had no additional effect. Keene (11) reported that the shrinkage of concrete cured in moist air for 7 days is greater than that of concrete allowed to dry immediately. California (Table 8 and Fig. 6) found substantially constant shrinkage in concrete that was moist cured for 7, 14, and 28 days before drying was started. In graded Ottawa sand mortars of varying richness (Table 2), specimens cured wet for 3 and 7 days developed equal amounts of

TABLE 5

EFFECT OF CHEMICAL ADMIXTURES ON SHRINKAGE SUMMARY OF MISCELLANEOUS TESTS[1]

Type of Admixture	Quantity per Sack of Cement		Maximum Size Aggregate (in.)	Cement Factor (sacks/cu yd)	Relative Shrinkage Control = 100
	Fl. Oz	Lb			
Hydroxylated carboxylate:					
a	2		1	7.0	100
	4		1	7.0	97
	3		1	7.0	93
	3		1	7.0	94
	2		1	7.0	104
	4		1	7.0	98
		0.5	1	7.0	104
		1.0	1	7.0	104
	2		1	7.0	104
	4		1	7.0	104
	2		1½	5.5	90
	1		1½	6.0	91
	2		1½	6.0	87
	3		1½	6.0	85
b	2.75		1	6.0	143
c	2.5		1	6.0	141
	4.5		1	6.0	161
Lignosulfonate:					
a		0.125	1	7.0	113
		0.25	1	7.0	113
		0.125	1	7.0	110
		0.25	1	7.0	114
		0.125	1	7.0	111
		0.25	1	7.0	114
		0.25	1½	5.5	106
b	4		1	7.0	106
	8		1	7.0	113
	4		1	7.0	106
	8		1	7.0	108
	3.75		1½	6.0	103
	7.5		1½	5.5	106
	7.5		1½	6.0	113
	11.25		1½	6.0	120
c		0.25	1	7.0	137
		0.25	1	7.0	138
		0.15	1	7.0	126
		0.30	1	7.0	144
		0.125	1	7.0	123
		0.25	1	7.0	144
		0.25	1½	6.0	133
		0.20	1	6.0	130
d	4.5		1	7.0	103
	9		1	7.0	106
	3		1½	5.5	97
e		0.125	1	7.0	141
		0.25	1	7.0	155
		0.125	1	7.0	141
		0.25	1	7.0	156
f		0.15	1	7.0	109
		0.30	1	7.0	119
		0.25	1	5.0	101
		0.25	1	6.0	98
		0.25	1	7.0	105
		0.25	1	8.0	99
		0.15	1	7.0	106
		0.30	1	7.0	114
		0.25	1½	5.5	102
		0.25	1	6.0	120
g		0.15	1	7.0	119
		0.30	1	7.0	133
h		0.15	1	7.0	107
		0.30	1	7.0	118
		0.20	1	6.0	106
i	4		1	7.0	111
	8		1	7.0	121
j		0.25	1½	5.5	113
k		0.125	1½	5.5	105
l	6		1½	5.5	131
Table sugar	0.3[2]		1½	5.5	100
unclassified					
a	0.5		1	7.0	94
	1.0		1	7.0	94
b	1.0		1	7.0	100
	2.0		1	7.0	100
c	0.375		1	7.0	100
	0.75		1	7.0	103
	0.375		1	7.0	106
	0.75		1	7.0	103

---

[1] 5½- to 8-sack concrete, 3½- to 4-in. slump. Aggregate 1½-in. max. size (concrete wet sieved through ¾-in.) or 1-in. max. size; 3- by 3- by 10-in. prisms cured moist for 7 days, then dried for 14 days. Values are averages of 6 or more specimens from 2 or more batches mixed on different days. Values are relative to shrinkage of control concrete containing no admixture.

[2] Dry weight.

TABLE 6

EFFECT OF CALCIUM CHLORIDE ADMIXTURE ON SHRINKAGE[1]

Type	Chloride Radical (%)[2]	Slump (in.)	Air (%)	Unit Wt. (pcf)	Water-Cement Ratio (% by wt.)	Cement Factor (sacks /cu yd)	Flow (%)
Fresh concrete[3]	0	3.7	1.6	150.8	0.502	7.01	
	0.04	3.5	1.8	150.5	0.493	7.01	
	0.08	3.5	1.6	150.9	0.492	7.02	
	0.16	3.6	1.9	150.4	0.479	7.01	
	0.32	3.6	2.1	150.5	0.493	7.00	
	0.64	3.5	2.3	150.5	0.478	7.02	
	1.28	3.5	2.6	149.6	0.492	6.97	
Fresh mortar[4]	0				0.363		104
	0.04				0.363		107
	0.08				0.363		106
	0.16				0.363		109
	0.32				0.365		109
	0.64				0.367		108
	1.28				0.390		110

[1] Figure 2 shows shrinkage results.

[2] By weight of cement.

[3] Average of 3 batches; Test Method No. Calif. 530.

[4] Average of 3 batches; 1:2 graded Ottawa sand mortar; Test Method No. Calif. 527.

TABLE 7

EFFECT OF TRIETHANOLAMINE ADMIXTURE ON SHRINKAGE[1]

Type	Triethanolamine (%)[2]	Slump (in.)	Air (%)	Unit Wt. (pcf)	Water-Cement Ratio (% by wt.)	Cement Factor (sacks /cu yd)	Flow (%)
Fresh concrete[3]	0	3.5	1.4	151.2	48.5	7.01	
	0.03	3.5	2.0	150.6	48.0	7.03	
	0.06	3.5	2.2	150.4	48.7	6.98	
	0.12	3.3	2.4	150.0	48.1	6.99	
	0.24	3.4	2.6	149.8	48.4	7.01	
	0.48	3.4	2.9	149.4	46.3	6.99	
Fresh mortar	0				0.371		101
	0.03				0.371		101
	0.06				0.371		103
	0.12				0.371		104
	0.24				0.371		104
	0.48				0.371		105

[1] Figure 3 shows shrinkage results.

[2] By weight of cement.

[3] Average of 3 batches; Test Method No. Calif. 530.

[4] Average of 3 batches; 1:3 graded Ottawa sand mortar; Test Method No. Calif. 527.

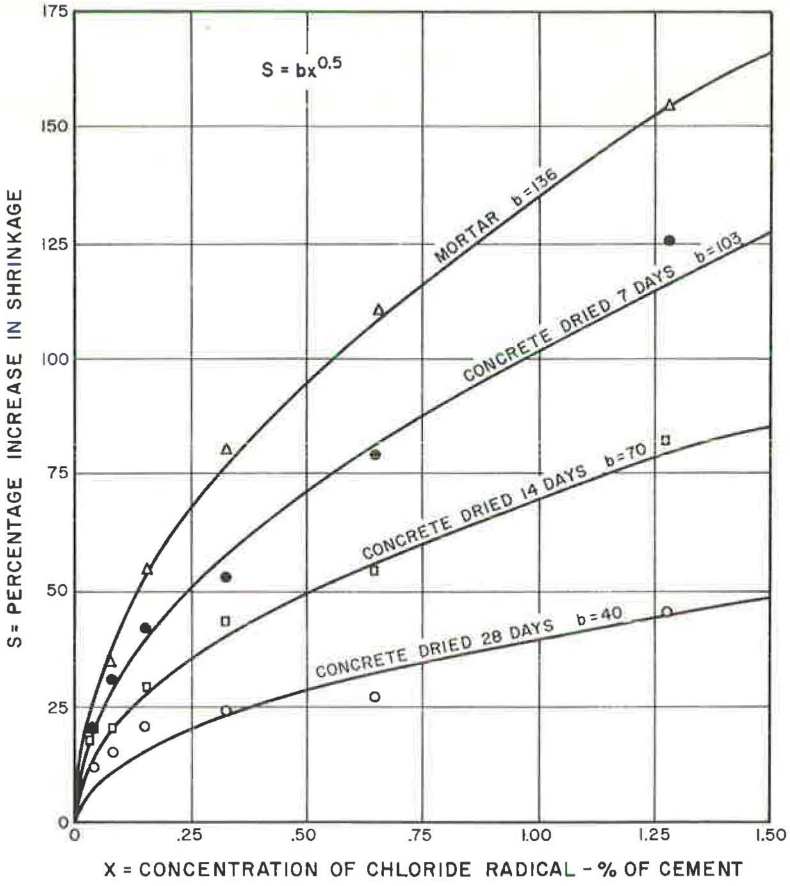

Figure 2. Effect of $CaCl_2$ admixture on shrinkage of mortar and concrete.

shrinkage but shrank slightly less than those cured wet for only 2 days. An analysis of variance shows the difference between 2 and 3 days of curing to be highly significant.

Powers (27) states that, from fundamental considerations, prolonging the curing period should increase the amount of shrinkage of cement paste. He states that the effect of curing of concrete may be expected to be more complicated and that prolonged curing makes paste more prone to crack when severely restrained. If cracking of paste relieves stresses around aggregate particles, the over-all shrinkage may thereby be diminished.

It appears that, given a period of moist curing adequate to develop reasonable strength, additional moist curing is relatively unimportant in the control of shrinkage.

Length of Drying Period

All tests made by California show that under uniform drying conditions, shrinkage is proportional to the logarithm of time of drying up to the age at which the concrete approaches moisture equilibrium with the environment. Further shrinkage at later ages is believed to be due to carbonation.

For specimens 3 by 3 in. in section dried under California standard laboratory conditions (temperature, 73.4 ± 3F; relative humidity, 50 ± 4 percent; evaporation from atmometer, 3 ± 0.5 ml per hr), shrinkage due to drying appears to stop after about 32 weeks. Figures 6, 7, and 8 show the relationship of shrinkage to period of drying.

Combined Effect of Unfavorable Factors

Powers (27) pointed out that the cumulative effect of individual factors that increase drying shrinkage can be very large. His calculations were made on the assumption that the combined effect is the product of the individual effects and is, therefore, much greater than if they were simply additive. Although the assumption is logical, Powers offered no data to support it.

California has obtained data that support the theory that the cumulative effect is the product, not the sum, of individual effects (Table 9). The data show the shrinkage produced by two aggregates, each used with and without a chemical admixture. The data are used to compute the shrinkage for aggregate B with the admixture, first on the assumption that the effects are additive (Item 4) and second, that they are factors to be multiplied together (Item 7). Item 8 shows the results as measured (Table 17 gives additional data). Simple addition consistently underestimates the shrinkage, whereas multiplication yields a close estimate of the observed shrinkage.

The effect of characteristics of materials has been discussed in preceding sections. In addition, there are several construction practices that can increase

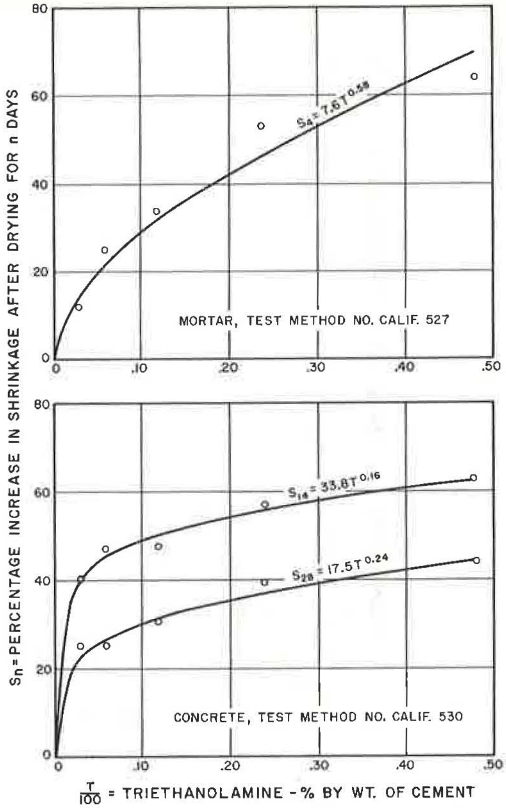

Figure 3. Effect of triethanolamine admixture on shrinkage of mortar and concrete.

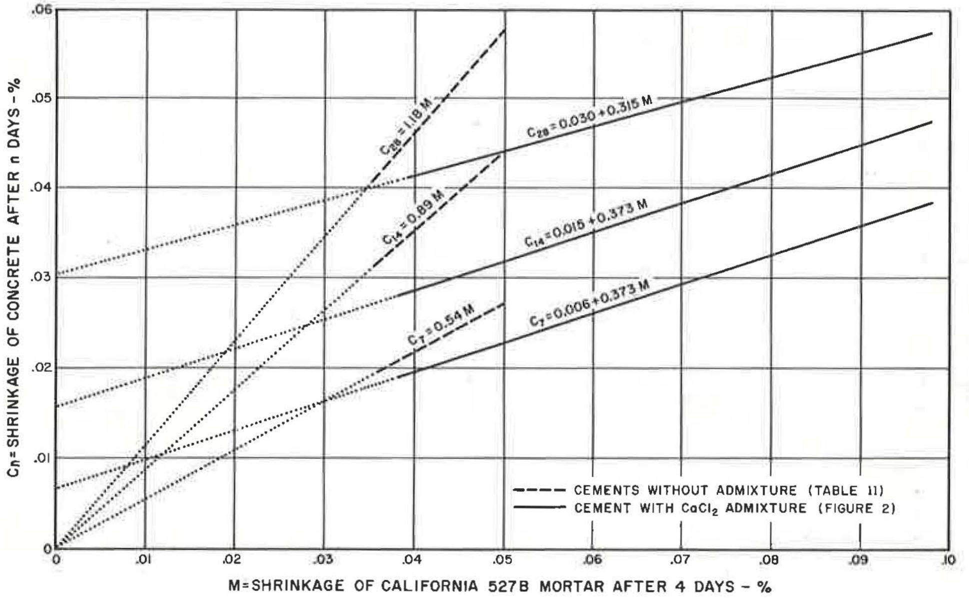

Figure 4. Effect of $CaCl_2$ on shrinkage relationship of mortar and concrete.

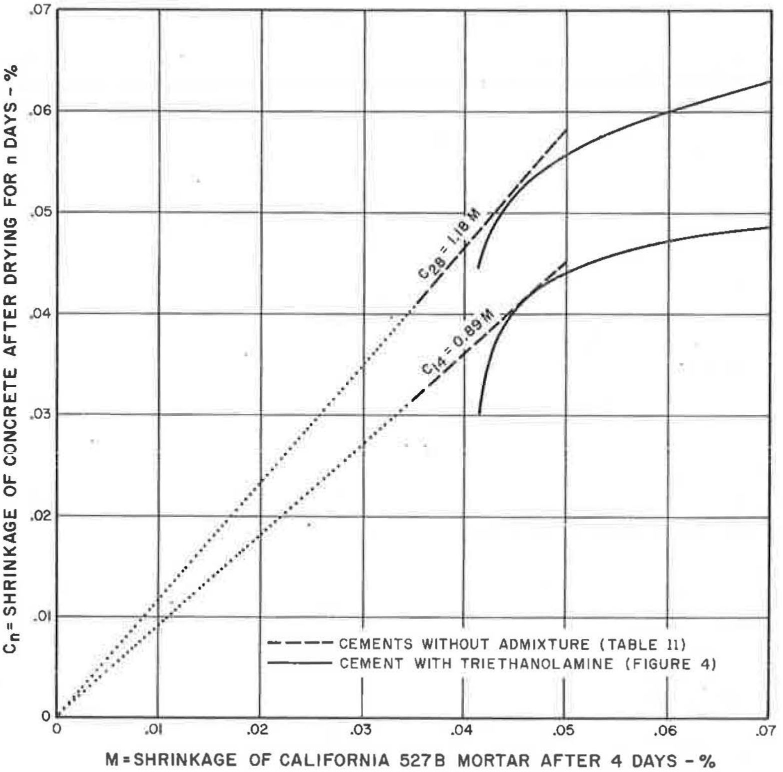

Figure 5. Effect of triethanolamine on shrinkage relationship of mortar and concrete.

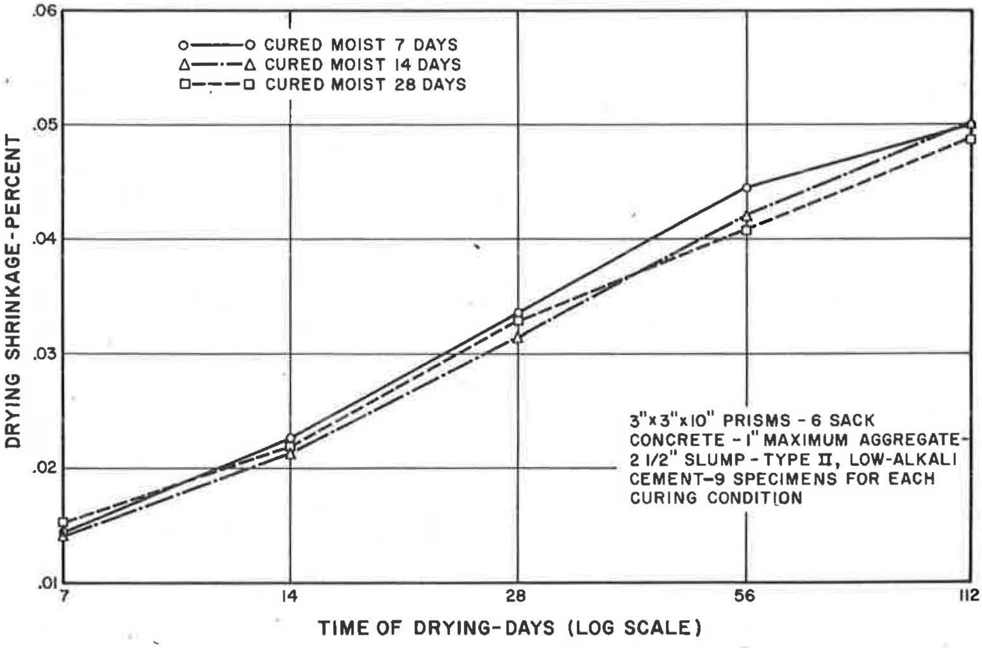

Figure 6. Effect of duration of preliminary moist curing on shrinkage.

TABLE 8

EFFECT OF DURATION OF PRELIMINARY MOIST CURING ON SHRINKAGE OF CONCRETE[1]

Fresh Concrete[2]				Period of Drying (days)	Shrinkage[3] (%)		
Slump (in.)	Air (%)	W/C by Wt.	Cement Factor (sacks)		7-Day Moist Curing	14-Day Moist Curing	28-Day Moist Curing
2.6	1.8	0.512	6.02	7	0.0144	0.0140	0.0153
				14	0.0226	0.0212	0.0218
				28	0.0337	0.0314	0.0328
				56	0.0447	0.0423	0.0412
				112	0.0498	0.0506	0.0492

[1] Type II, low-alkali cement; 1-in. maximum aggregates from American River; nine 3- by 3- by 10-in. prisms (3 selected from each curing period) molded from each batch.
[2] Average of three batches.
[3] Average of nine specimens.

shrinkage by reason of an increase in water demand. From Figure 7 of USBR Concrete Manual (9), assuming a cement factor of 6 sacks per cubic yard, it is estimated that an increase of 50 lb of water per cubic yard increases shrinkage 160 millionths, or 30 percent. Using this basis the following possible increases in shrinkage result.

Temperature. --Figure 99 of the USBR Concrete Manual (9) shows that 13 pcy of additional mixing water are required for a 20F increase in temperature of concrete as it is mixed. The equivalent increase in shrinkage is 8 percent.

Slump. --Table 3 of ACI Standard 613 (16) shows that the use of a slump of 6 to 7 in. requires 17 pcy more water than does a slump of 3 to 4 in. The equivalent increase in shrinkage is 10 percent.

Maximum Size of Aggregate. --Table 3 of ACI Standard 613 (16) indicates that the use of ¾-in. aggregate requires 42 pcy more water than does 1½-in. aggregate. The equivalent increase in shrinkage is 25 percent.

Time of Mixing or Agitation. —After satisfactory mixing has been achieved, additional mixing, or prolonged agitation produces additional aggregate fines and increases temperature of the concrete. Both effects tend to increase the water demand for a given slump. The specific increase is a variable depending on characteristics of the aggregates and other factors. For an unnecessary delay of 30 min in discharging from a transit mixer, a reasonable assumption is that the water demand may be increased by about 15 pcy, which is equivalent to a 10 percent increase in shrinkage.

In Table 10, the cumulative effect on shrinkage of eight individual factors is computed as the product of the individual effects. Justification for the use of the values assigned to each factor has been previously discussed, and they are believed to be conservative. The table indicates that a fourfold increase in shrinkage is a distinct possibility in many projects. The first four factors listed are those subject to job control under most specifications. The combined effect of the four departures from the best practice is an increase of 64 percent in shrinkage. Although important in many types of work, this increase in shrinkage is but a small part of that which may result from a poor selection of the constituent materials of the concrete.

Analysis of Laboratory Studies

Although methods employed by different investigators varied considerably, in general, mortar specimens were prisms of 1 by 1 to 2 by 2 in. in section. Concrete specimens have ranged from 3 in. in diameter to 4 by 5 in. in section with occasional

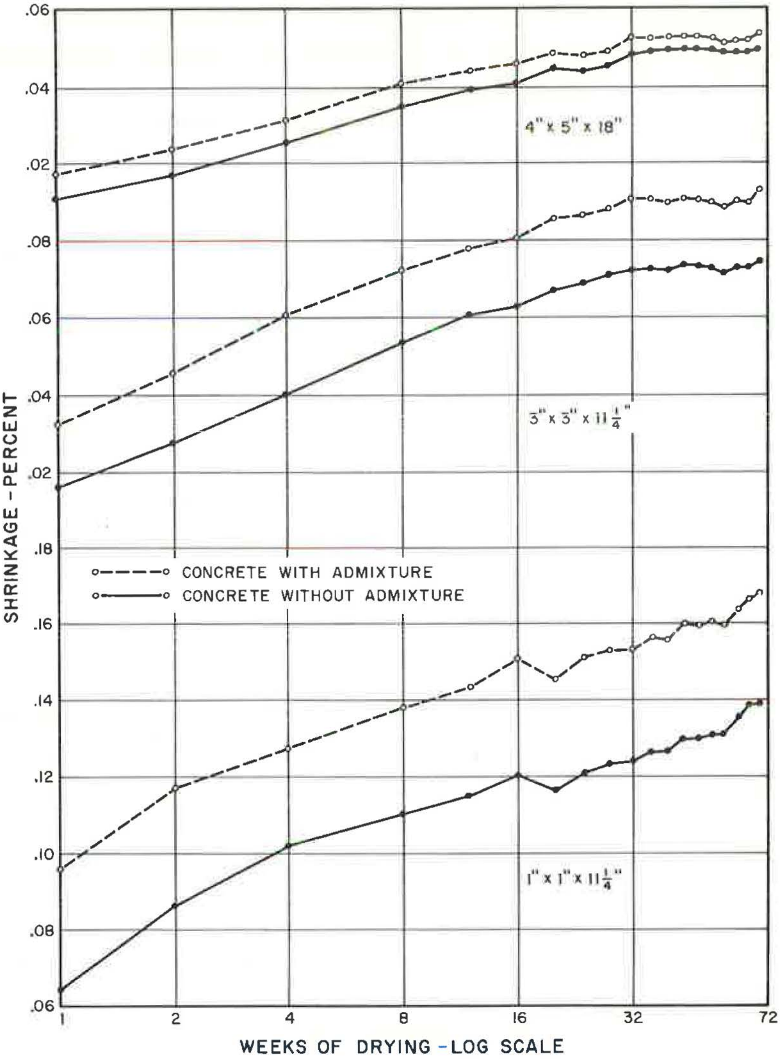

Figure 7. Increase in shrinkage with length of drying period.

tests on larger specimens. Drying has been accomplished at room temperature or in an atmosphere controlled at about 73F and 50 percent relative humidity. Except for California, no laboratory appears to have given consideration to the effect of air movement on the rate of evaporation of water from concrete.

Few, if any, have questioned that a prism of concrete 4 in. square provides a significant index of shrinkage. A number have used specimens 3 in. square or 3 in. in diameter. Some have questioned the significance of specimens containing mortar only in evaluating shrinkage of concrete even though the variables under study do not include coarse aggregate.

California compared the shrinkage of eight Type II, low-alkali cements in mortar and concrete with results as summarized in Table 11. It was found that the shrinkage of the concrete could be predicted from the mortar test result, using a linear equation represented by a curve passing through the origin, with a standard error of estimate of less than 10 percent (Table 16 gives additional data). Data available from two other laboratories, involving cements of other types as well, indicated similar precision of

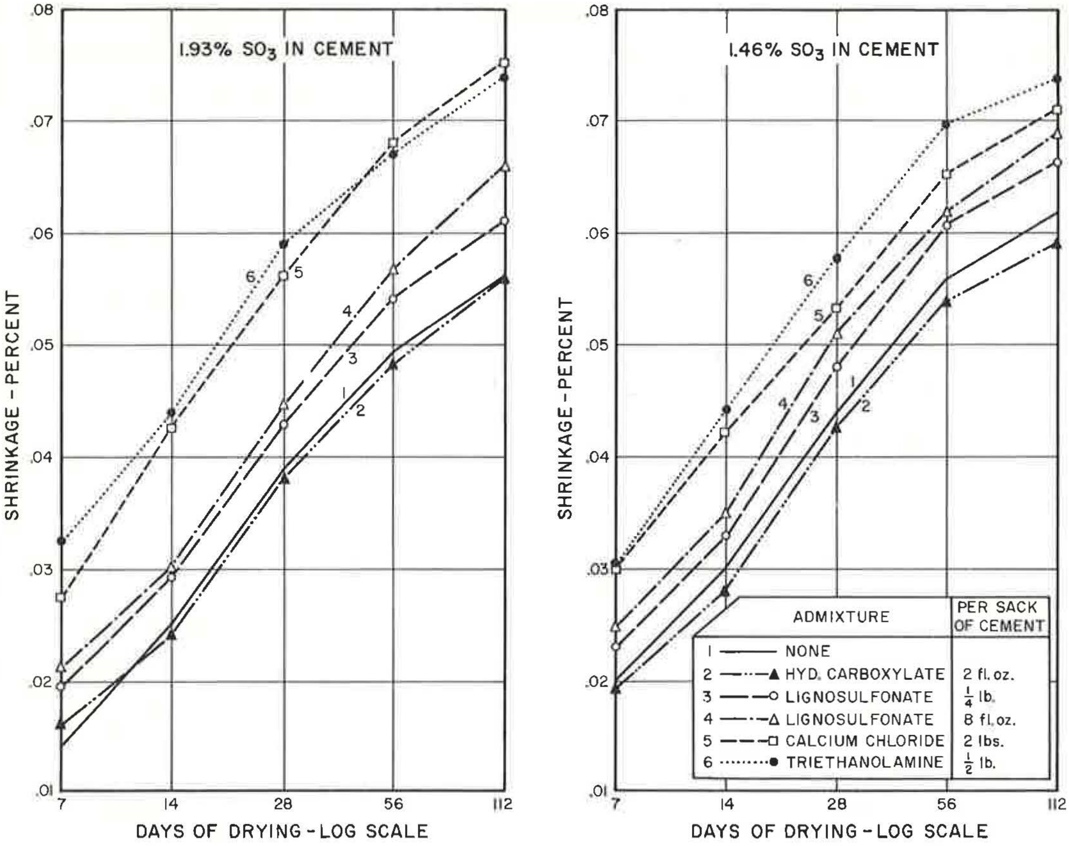

Figure 8. Increase in shrinkage with time of drying.

prediction. The equations were of the form

$$Y = bX \tag{3}$$

in which b varies with the time of drying concrete.

On the other hand, California found that the concrete-to-mortar relationship cannot be expressed satisfactorily by Eq. 3 if additions or admixtures that react chemically with the cement are used. For calcium chloride, the equation, although linear, is

$$Y = bX + a \tag{4}$$

in which the value of a is of considerable magnitude (Fig. 4).

For triethanolamine, a curvilinear relationship was found between the shrinkage of concrete and that of mortar (Fig. 5). It appears therefore, that mortar tests are satisfactory for comparing the shrinkage of cements unless they have been interground with reactive additions. When cements contain reactive additions or when admixtures are used, shrinkage should be determined by means of tests of concrete rather than mortar unless the concrete-to-mortar relationship has been established by prior tests for each addition or admixture.

Objections have been raised to the use by California of Test Method No. Calif. 530 for determining the effect of admixtures on shrinkage. One objection is that the expression of the effect on shrinkage in terms of a percentage of the control concrete (without the admixture) is not realistic because of the small base values involved and that the use of absolute numerical differences in shrinkage is preferable.

The following test data are presented in answer to this objection. Table 9 shows

TABLE 9

EFFECT OF INDIVIDUAL SHRINKAGE FACTORS ON CUMULATIVE SHRINKAGE

Tests by Test Method No. Calif.530 except aggregates were from different sources. Lignosulfonate water-reducing admixture at 0.25-lb. per sack of cement.

Properties of Fresh Concrete
(Average of 4 batches)

	Slump, Inches	Air %	W/C By Wt.	Unit Wt. Lbs./CF	Cement Factor Sks/CY
Aggregate A					
Control	3.5	1.8	.445	148.0	7.06
With Admixture	3.4	5.0	.388	144.4	7.02
Aggregate B					
Control	3.8	1.5	.468	150.5	7.05
With Admixture	3.6	4.4	.406	148.0	6.99

Shrinkage, Percent, After Number of Days Shown
(Average of 12 specimens from 4 batches)

	Aggregate A					
	Control			Admixture		
	7	14	28	7	14	28
Shrinkage	.0170	.0230	.0293	.0240	.0319	.0397
Numerical increase				.0070	.0089	.0104
% of Control	100	100	100	141	137	135
	Aggregate B					
Shrinkage	.0305	.0443	.0594	.0455	.0611	.0760
Numerical increase				.0150	.0168	.0166
% of Control	100	100	100	149	138	128

	Days of Drying			
	7	14	28	Avg.
(1) Shrinkage, Aggregate A	.0170	.0230	.0293	
(2) Increased shrinkage due to admixture, Aggregate A	.0070	.0089	.0104	
(3) Shrinkage, Aggregate B	.0305	.0443	.0594	
(4) Computed shrinkage of Aggregate B with admixture, (2) + (3)	.0375	.0532	.0698	
Average of Drying Periods				.0535
(5) Relative Shrinkage, admixture to Control, Aggr. A	1.41	1.37	1.35	
(6) Relative Shrinkage, Aggregate B to Aggregate A	1.80	1.92	2.03	
(7) Computed Shrinkage of Aggregate B with admixture, (5) x (6) x (1)	.0430	.0609	.0800	
Average of Drying Periods				.0613
(8) Measured Shrinkage of Aggr. B with admixture	.0455	.0611	.0760	
Average of Drying Periods				.0609

TABLE 10

CUMULATIVE EFFECT OF ADVERSE FACTORS ON SHRINKAGE[1]

Factor	Equivalent Increase in Shrinkage (%)	Cumulative Effect
Temperature of concrete at discharge allowed to reach 80F, whereas with reasonable precautions, temperature of 60F could have been maintained	8	1.00 × 1.08 = 1.08
Used 6- to 7-in. slump where 3- to 4-in. slump could have been used	10	1.08 × 1.10 = 1.19
Use of ¾-in. max size of aggregate under conditions where 1½-in. size could have been used	25	1.19 × 1.25 = 1.49
Excessive haul in transit mixer, too long a waiting period at job site, or too many revolutions at mixing speed	10	1.49 × 1.10 = 1.64
Use of cement having relatively high shrinkage characteristics	25	1.64 × 1.25 = 2.05
Excessive "dirt" in aggregate due to insufficient washing or contamination during handling	25	2.05 × 1.25 = 2.56
Use of aggregates of poor inherent quality with respect to shrinkage	50	2.56 × 1.50 = 3.84
Use of admixture that produces high shrinkage	30	3.84 × 1.30 = 5.00

[1] Based on effect of departing from use of best materials and workmanship.

that the numerical difference in shrinkage for a given admixture can vary widely depending on the characteristics of the aggregates used in the test concrete. On the other hand, if the performance of the admixture is expressed as a percentage relative to the control concrete, the rating is essentially constant regardless of the aggregate used in the test concrete.

FIELD PERFORMANCE

Carlson (28) and, later, Pickett (29) calculated the effect of size of member (or test specimen) on the rate of shrinkage by means of a coefficient of shrinkage diffusivity in a manner analogous to that used in studies of heat flow. Carlson concluded that, in large masses, moisture is fed toward the surface from the underlying concrete so as to prevent or delay substantial shrinkage except near the exposed surface, and that, under ordinary climatic conditions, the average shrinkage of structural members one foot or more in thickness probably would never approach that of small bars.

Keeton (30) found a curvilinear relationship between the surface-to-volume ratio of specimens of varying size and the amount of shrinkage at any time. California (Fig. 9) found that the relationship is presumably linear. It thus appears that size of test specimen per se is of little practical importance, and that equal values of shrinkage can be obtained from different sizes of specimen if drying times are suitably varied. Long periods of drying introduce the hazard of variations in test conditions. This suggests that the specimen should be of the smallest cross-section suitable for the maximum size of aggregate involved in the concrete.

TABLE 11

CONCRETE AND MORTAR SHRINKAGE TESTS OF EIGHT TYPE II, LOW-ALKALI CEMENTS

Property	Mortar[1] 4 Days	Concrete[2]		
		7 Days	14 Days	28 Days
Cement No.				
1	0.0374	0.0219	0.0365	0.0488
2	0.0448	0.0233	0.0365	0.0487
3	0.0386	0.0221	0.0363	0.0598
4	0.0384	0.0226	0.0354	0.0586
5	0.0462	0.0273	0.0470	0.0600
6	0.0394	0.0186	0.0308	0.0544
7	0.0462	0.0228	0.0371	0.0599
8	0.0471	0.0252	0.0388	0.0636
Value of b[3]				
Std. error of est.				
(S_{yx}):		0.54	0.89	1.18
Numerical		0.0021	0.0038	0.0041
Percent		9	9	8

[1] Mortar tests by Test Method No. Calif. 527; each value is average of 12 specimens from 3 batches.

[2] Concrete tests by Test Method No. Calif. 530 except cement factor is 6 sacks per cu yd, and no admixtures were included; each value is average of 12 specimens from 4 batches.

[3] In equation Y = bX.

[4] $\frac{S_{yx}}{\text{Avg. obs. concrete shrinkage}} \times 100.$

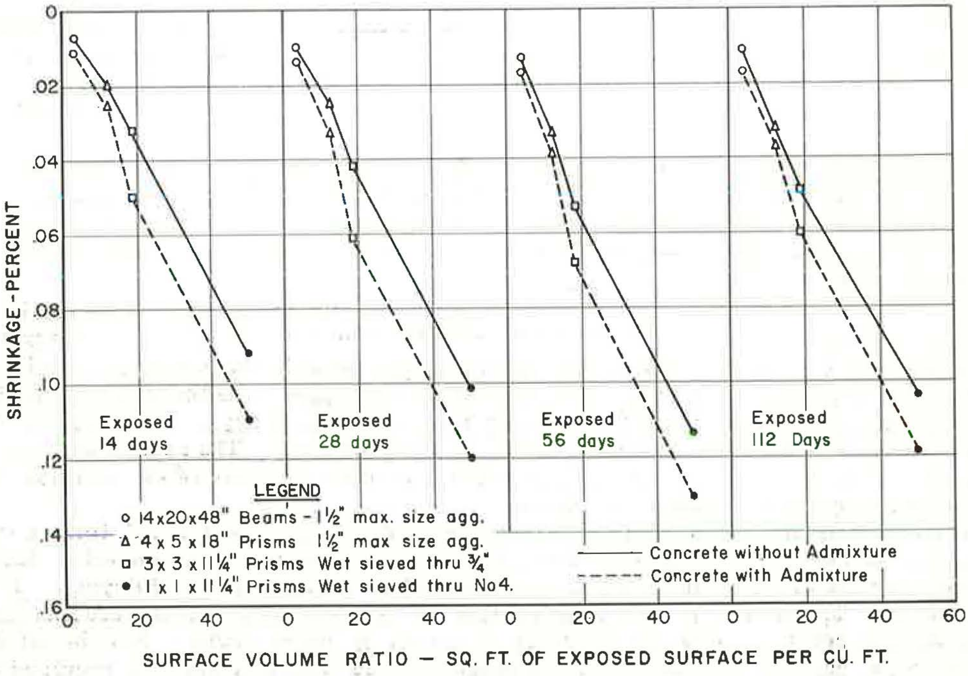

Figure 9. Relationship between shrinkage and surface-volume ratio; specimens exposed outdoors.

An objection has been raised to the use by California of Test Method No. Calif. 530 for determining the effect of admixtures on shrinkage on the ground that a drying period of only 14 days is too short to establish relationships that are significant in terms of long-time field performance. Answers to the objection are provided by the following experiment and by results presented later.

California investigated the effect of a chemical admixture on shrinkage of 14- by 20- by 48-in. beams that were exposed out of doors at Sacramento. The admixture under study was the same as that in Table 9. It was tested with a third aggregate (also in accordance with Test Method No. Calif. 530) and was found to produce a relative shrinkage of 144 percent. The average increase in shrinkage at 14 days as determined by the use of three aggregates is 40 percent. Outside exposure of the beams was started in August 1960. Shrinkage took place for 2 months after which the beams began to lengthen. Shrinkage resumed in the early spring and continued until November of that year. Table 12 gives the relative increase in drying shrinkage due to the admixture through three drying seasons. During the last half-year of the second drying season the admixture caused an average increase of 32 percent in shrinkage. At the end of the third drying season, the increase was reduced to 23 percent. The increase is less than 40 percent as found by the laboratory tests of prisms. It should not be inferred, however, that a test drying period of 14 days does not afford a realistic measure of the effect of admixtures. It is to be expected that the performance of admixtures in service will vary, depending on weather conditions and size of member. No single laboratory test can be expected to predict service performance under all conditions. For specification purposes, the test is as significant as for one of a longer period. Furthermore, early shrinkage may be of greater importance than final shrinkage. The test has the great advantage that the time elapsed in conducting it is less than 28 days. It is feasible to use it, therefore, for control testing of deliveries to the work. The results demonstrate that Test Method No. Calif. 530 provides a reliable measure of the performance of concrete subjected to exterior exposure.

Figure 10 uses the data of Figure 8 to show the change taking place with time of drying in the ratio of shrinkage produced by admixtures to that of control concrete. Ratios at 7 days are shown to be much higher than those after longer periods of drying. For admixtures 2, 3, and 4 (Fig. 10), representing the set-retarding, water-reducing type, ratios determined at 14 days are not changed greatly by longer drying. For the straight accelerators, calcium chloride and triethanolamine, 28 days of drying are required to obtain reasonably stable ratios. The order of rating the admixtures is essentially the same at all drying periods. Because water-reducing admixtures, if they contain ac-

TABLE 12

EFFECT OF A CHEMICAL ADMIXTURE[1] ON SHRINKAGE OF A 14- BY 20- BY 48-IN. BEAM EXPOSED OUTDOORS

Date	Length of Exposure (No. of weeks)	Increase in Shrinkage[2] (% of control concrete[3])[4]
Oct. 1960	8	36
Nov.	12	37
Dec.	16	44
Dec.	20	50
Jan. 1961	24	59
Feb.	28	39
Mar.	32	48
Apr.	36	35
May	40	36
June	44	31
July	48	33
Aug.	52	32
Sept.	56	31
Oct.	61	31
Nov.	65	32
Dec.	69	33
Feb. 1962	78	36
May	93	33
Sept.	111	22
Dec.	123	23

[1] 6-sack concrete, 3- to 4-in. slump, 1½-in. max. size aggregate; moist-cured 7 days, then exposed outdoors at Sacramento, Aug. 1960.

[2] Due to admixture.

[3] Without admixture.

[4] Average of measurements at center and surface.

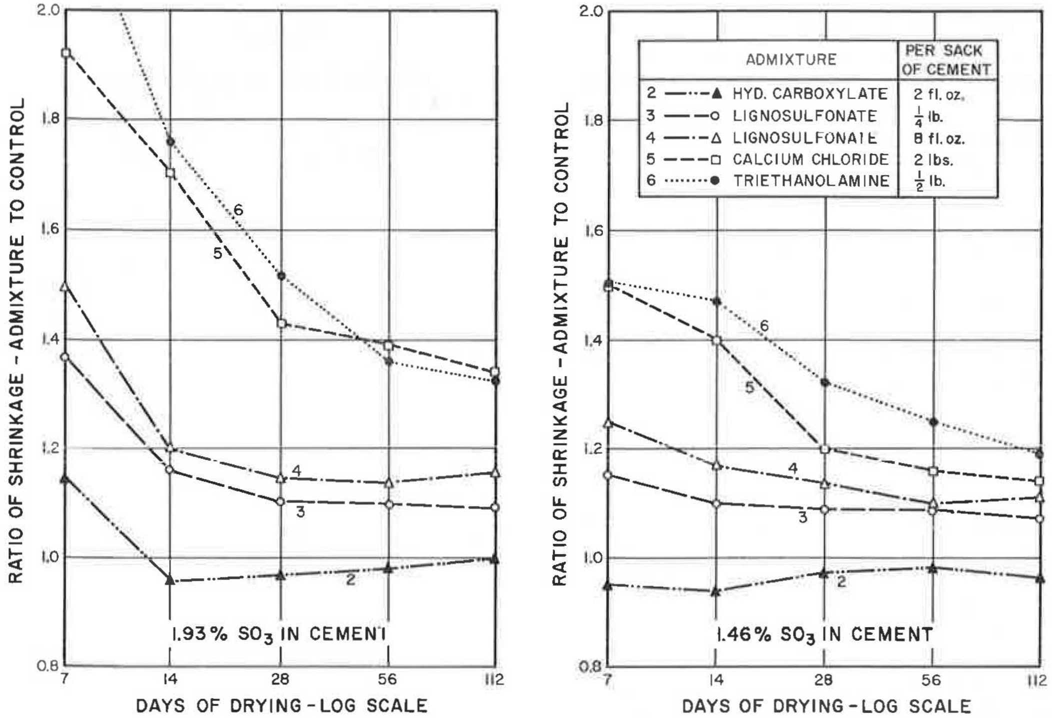

Figure 10. Change in relative shrinkage with drying time.

celerators, contain relatively small amounts of them, it is not seriously unrealistic to evaluate their performance with respect to shrinkage after 14 days of drying.

The data presented thus far indicate that the effect of the cement on shrinkage can be measured satisfactorily in 1- by 1- by 10-in. bars of graded Ottawa sand mortar if it contains no addition. Tests of admixtures should be made in concrete with aggregates graded up to ¾ in. or larger. The test specimen may be as small as 3 by 3 by 10 in. and the period of drying may be as short as 14 days.

Comparisons of aggregates should be made with specimens large enough for concrete of the largest maximum size under consideration. When specimens larger than 3 by 3 in. in section are required, the period of drying should be extended to yield numerical results of comparable magnitude. Test specimens 4 by 5 in. in cross-section have been used with 1½-in. maximum size aggregate. Six-inch diameter cylinders have also been used. In either case, a drying period of 28 days has been found to produce shrinkage comparable in magnitude to that obtained by drying 3- by 3-in. specimens for 14 days.

Pavement Performance Related to Shrinkage

California has investigated the surface contour of pavements by means of a profilograph. The results show a pronounced tendency for slabs to curl upward at the ends. Curling is due to unequal moisture and temperature distribution from top to bottom. The upper portion of a pavement is nearly always drier than the bottom. Upward curling due to a moisture differential may be offset wholly or partially in the afternoon by a higher temperature at the top than at the bottom. The temperature effect seldom produces a downward curvature. At night, a reversal in temperature distribution adds to the curling due to uneven moisture distribution. During the greater part of the time, pavement slabs are curled upward. Figure 11 shows pavement slabs as they appeared shortly after dawn. Surface water has drained from the slab ends which have dried sufficiently to show a visible contrast.

Figure 11. Curled pavement slabs.

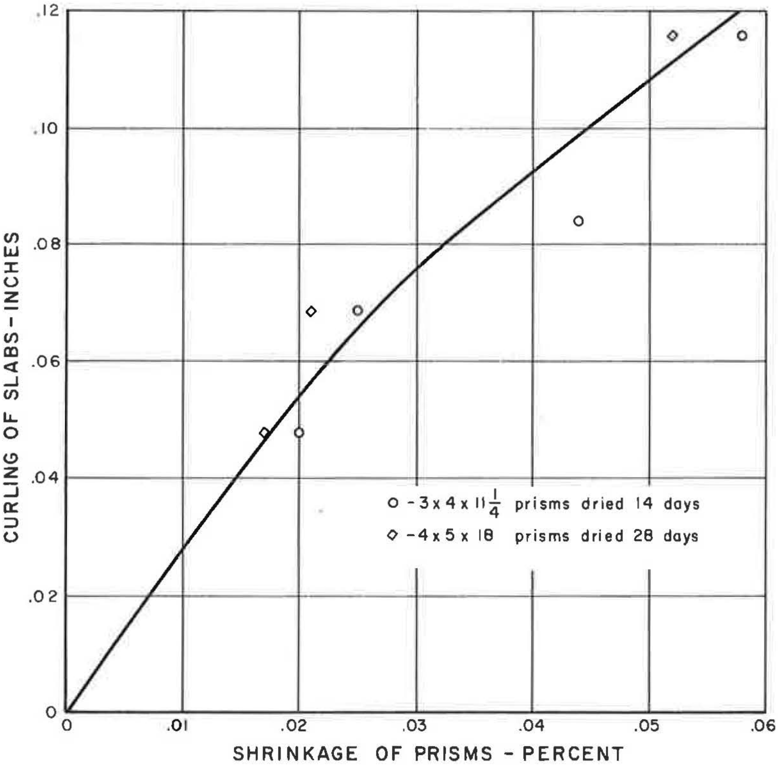

Figure 12. Slab curling related to concrete shrinkage.

Hveem (3) found that deflections under a heavy axle load were equal at the end and the middle of slabs when they were essentially flat; that is, in contact with the subgrade at all points. When the slabs were curled upward, deflections at the ends were four times as great as at the middle.

Although it appears to be axiomatic that concrete of greater shrinkage should produce greater curling, California has obtained direct evidence that this is true (Fig. 12). Also, it has obtained evidence that if curling is sufficiently severe, deflections under traffic result in a high incidence of slab cracking.

TABLE 13

SHRINKAGE AND CURLING IN KANSAS TEST CEMENTS

Cement	Shrinkage (%)	Curling (in./slab)
19A	0.022	0.139
19B	0.027	0.147
19C	0.029	0.182

As a part of the long-time study of the performance of portland cement in concrete, the Kansas Highway Department constructed a four-lane divided highway $4\frac{1}{2}$ miles long near Topeka. Among the variables studied were three cements, LTS 19A, 19B, and 19C, representing "old-fashioned," "modern coarse ground," and "modern" cements, respectively. Each cement was used in one-third of the experimental sections which were 1,000 ft in length. Results of shrinkage tests for 2- by 2- by 11-in. prisms of concrete containing the cements after drying for 1 month are given in Table 13.

In 1956, when the pavement was 7 years old, California obtained profilograms of the pavement using a 25-ft manual profilograph. The average amount of permanent curling of slabs containing each cement was measured from the profilograms (Table 13).

The order of the cements with respect to slab curling is the same as the order in the shrinkage tests. To this extent, the laboratory tests for cement are found to be significant with respect to pavement performance.

One of the most productive comparisons of pavement performance with laboratory shrinkage data is found in the results obtained when calcium chloride was used as an admixture. Laboratory tests of calcium chloride by Calif. 530-A are shown in Figures 2, 8, and 10.

Depending on the cement used, calcium chloride at the rate of 2 lb per sack of cement may be expected to increase the shrinkage of concrete by 40 to 70 percent as measured by Test Method No. Calif. 530. The effect of using 1 lb per sack is about three-fourths as great. Short sections of pavement have been constructed frequently with calcium chloride in California for purposes of accelerating strength gain. Although many of such sections were less than 100 ft in length, records of a few were found that were at least several hundred feet in length, that were constructed concurrently with other pavement, and that were of an age suitable for comparative purposes.

Table 14 summarizes data of four such projects with respect to slab curling, as measured from profilograms, and the development of cracking.

In the Merced County project, 77 percent of the slabs in the outer, or driving, lane containing calcium chloride have cracked transversely at about the midpoint. The amount of curling in these half-slabs averages 0.10 in. It is assumed that before the slabs cracked, the curling was 0.20 in. which is the value shown in the table. This section is noticeably "rough riding." Figure 13 shows profilograms of the Merced County pavement. This figure also shows the method of measuring slab curling. Figure 14 shows the section containing calcium chloride. Although both lanes contain calcium chloride, cracking has developed only in the outer lane which receives heavy truck traffic on this 4-lane divided highway.

Profilograph data as summarized in Table 13 show that the use of calcium chloride has increased the amount of curling in pavement slabs by 50 percent as an average of the four projects. As a consequence of the increased curling, the riding qualities of the pavements have been impaired. In the lanes receiving heavy traffic, a substantial increase in cracking has occurred. The results, therefore, provide striking evidence that Test Method No. Calif. 530 rates the effect of admixtures on shrinkage in a highly significant manner.

TABLE 14

EFFECT OF CALCIUM CHLORIDE ADMIXTURE ON PERFORMANCE OF PAVEMENTS[1] IN CALIFORNIA

Location (county)	Date Constr.	Age at Survey (yr)	Length of Sections (ft)	Section[2] Contains $CaCl_2$	$CaCl_2$ per Sack of Cement (lb)	Avg. Curl per Slab[3] (in.)	Slabs (%)			
							With Transverse Cracks	With Longitudinal Cracks	With Partial Cracks	With Corner Breaks
Merced	1955	6	840	Yes	1	0.20	77	29	29	2.0
				No[4]	-	0.11	13	0	20	2.0
Sacramento-San Joaquin	1957	4	9,800	Yes	2	0.09	14	0	0	1.8
				No[5]	-	0.06	8	0	0	0.1
Santa Barbara	1956	5	650	Yes	2	0.13	19	0	13	0
				No[4]	-	0.09	7	0	0	0
San Diego	1955	6	1,050	Yes	2	0.16	0	0	0	0
				No[4]	-	0.14	0	0	0	0

[1] All slabs 15 ft between weakened plane joints. Surveys made in outer, traveled lane of divided highways, except in San Diego which is an inner, passing lane.

[2] Length of section not containing $CaCl_2$ approximately equal to that with $CaCl_2$.

[3] From profilograms.

[4] In same lane and adjacent to section with $CaCl_2$.

[5] Opposite section with $CaCl_2$.

Highway Structure Performance Related to Shrinkage

In a preceding section, reference was made to the shrinkage of two model beams that were exposed out of doors at Sacramento. Weather locations at other sites may be expected to result in different rates and degrees of drying. To evaluate conditions at other locations, humidity measurements have been made periodically in the concrete of eight existing structures in California. The locations were selected to represent elevations from 200 ft below to 6,000 ft above sea level, and from moist coastal to arid desert climates.

During July 1960, two 1½-in. holes were drilled in the side of an interior girder of each of six of the selected structures. One structure was of box girder design and the holes were drilled in an exterior side facing north. In the remaining structure, of flat slab design, the holes were drilled in the east face. Holes were drilled to depths of

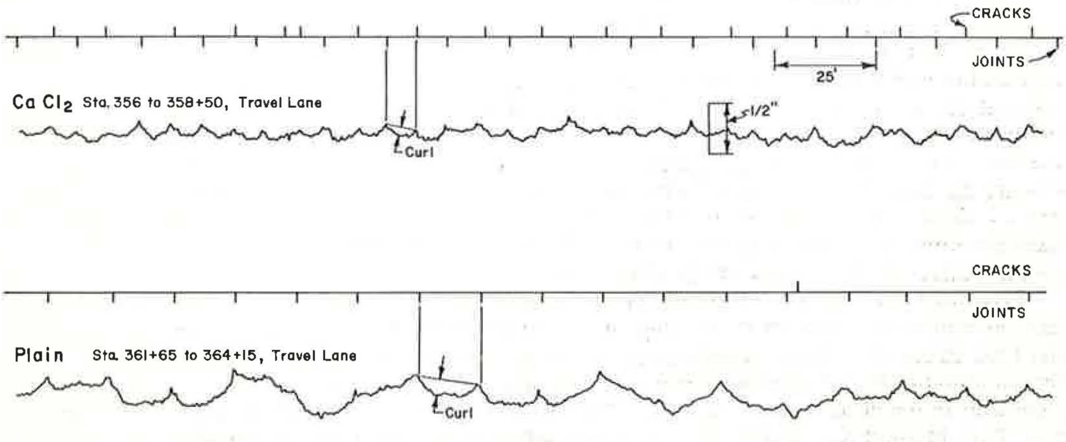

Figure 13. Profiles of pavement, plain and with 1 percent $CaCl_2$.

Figure 14. Pavement containing $CaCl_2$, Merced County.

3 and 5 in. Brass tubes 1 in. shorter than the depth of the hole were cemented in place with epoxy adhesive, the tube ends being flush with the surface of the member. The outer end of the tube was threaded and the opening was closed with a screw cap. When tests for relative humidity within the concrete were made, the cap was removed and a humidity-sensing element connected to an electric hygrometer was inserted. The leads of the sensing element passed through a metal cap which replaced the one that was removed, and were sealed against leakage. Several readings were made until equilibrium was established, which required about one hour. The sensing elements were calibrated from time to time at 70F against saturated salt solutions which produce known relative humidities in the enclosed space above them. Measurements have been made five times at 3-month intervals with results as given in Table 15. This table shows the temperature within the test hole at the time of humidity measurement. There was considerable variation in temperature, but the observed humidity is corrected for temperature by means of a chart that is supplied with the instrument. Independent tests confirmed the accuracy of the manufacturer's charts.

The average of minimum relative humidities measured at the eight locations is 67 percent at the 3-in. depth and 70 percent at the 5-in. depth. Thus, the typical expectation in California is that concrete in structures will reach an average relative humid-

TABLE 15

TEMPERATURE AND RELATIVE HUMIDITY IN CONCRETE OF EXISTING BRIDGES IN CALIFORNIA

Location	Elevation Above Sea Level (ft)	Year Built	Date of Test (mo-yr)	At 2- to 3-In. Depth		At 4- to 5-In. Depth	
				Temp. (°F)	Rel. Hum. (%)	Temp. (°F)	Rel Hum. (%)
Br. No. 54-500R Mojave Desert near Victorville	3,000	1958	8-60	78	66	79	74
			1-61	53	67	55	74
			4-61	56	67	60	75
			8-61	87	67	88	76
			11-61	59	59[1]	58	72[1]
Br. No. 57-332L Southern Coast near San Diego	10	1956	8-60	74	80	71	81
			1-61	58	76[1]	54	79
			4-61	64	77	61	77[1]
			8-61	71	79	68	80
			11-61	68	77	68	79
Br. No. 36-65 Central Coast near Santa Cruz	10	1947	9-60	59	77	57	80
			1-61	49	76	46	77[1]
			4-61	54	78	50	79
			8-61	64	79	61	82
			11-61	54	75[1]	54	78
Br. No. 4-84R Northern Coast near Eureka	10	1929	9-60	54	84[1]	54	86
			1-61	48	86	48	86
			4-61	53	84[1]	53	84[1]
			8-61	57	86	60	87
			11-61	53	86	53	87
Br. No. 24-134 Central Valley near Sacramento	25	1959	9-60	87	74	82	77
			1-61	36	78	36	75
			4-61	57	77	55	76
			9-61	64	72[1]	61	75
			12-61	57	80	53	68[1]
Br. No. 19-106L Sierra Nevada Range near Kingvale	6,000	1959	8-60	57	70	57	77
			1-61	32	72	32	72
			4-61	45	72	47	69[1]
			8-61	68	70	67	75
			10-61	39	66[1]	38	69[1]
Br. No. 48-22 East of Sierras near Bishop	4,400	1949	8-60	83	50	80	60
			1-61	53	47[1]	52	51
			4-61	74	49	74	57
			8-61	85	48	85	57
			11-61	50	47[1]	52	50[1]
Br. No. 58-277 Imperial Valley near Salton Sea	-200	1950	8-60	88	58	88	67
			1-61	64	71	61	63[1]
			4-61	99	72	95	73
			8-61	101	67	103	71
			11-61	68	56[1]	68	63[1]

[1] Minimum observed at location.

ity of 70 percent. The only area where markedly greater drying was found was in the high desert area near Bishop. A review of weather records for the continental United States indicates that the test locations in California provide a fair representation of exposure conditions on a nationwide basis. Only in parts of Arizona is it indicated that drying conditions as severe as those at Bishop are encountered.

Exposure at Sacramento as indicated for Location 5 of Table 14 produces internal humidity that is close to the average expectation of 70 percent. The data of the model beams, presented in a preceding section, therefore, are of particular significance with respect to performance of structures subjected to exterior exposure. It was shown that laboratory shrinkage results after 14 days of drying provided significant data with respect to shrinkage of the model beams, and therefore, of exterior concrete in general.

Outward indications of the effects of excessive shrinkage of concrete in highway structures would be expected to be most evident in a bridge deck. Some cracking in the deck is to be expected with all concrete because of the relatively high percentage of steel. The evaluation of performance as affected by shrinkage in laboratory tests, therefore, must be by qualitative rather than strictly quantitative methods. Calcium chloride has been used in the decks of a number of California bridges. Abnormal amounts of cracking have been reported for the majority of structures containing calcium chloride. Figures 8 and 10 show that calcium chloride increases shrinkage when tested in the laboratory.

The decks of twin, parallel bridges over Webber Creek in El Dorado County were completed under contract in October 1962. The bridges are of composite reinforced concrete and steel girder design. Each is approximately 540 ft in length and is divided by expansion joints into four sections of approximately equal length. Four combinations of two cements and two aggregates were used in replicate in the eight-deck sections. One of the cements (No. 1) is high in C_3A and alkalies. Cement No. 2 is a Type II, low-alkali cement. The two aggregates produce concrete of greatly different shrinkage as measured by laboratory tests. Thus, concrete of four degrees of shrinkage was produced. The decks are instrumented to provide data of strain and deflection, which, together with visual observations over a period of years, will give information on the effect of concrete shrinkage of the performance of bridge decks of this design. Other data secured during the progress of the work now afford useful comparisons with the results of shrinkage tests described previously in this report.

TABLE 16

TESTS OF JOB-MIXED CONCRETE FOR WEBBER CREEK BRIDGES[1]

Aggregate	Cement[2]	Shrinkage (%)								Curl[5] (in.)
		3- by 3- by 11¼-In. Prisms[3]				4- by 5- by 18-In. Prisms[4]				
		7-Day Drying	14-Day Drying	28-Day Drying	56-Day Drying	7-Day Drying	14-Day Drying	28-Day Drying	56-Day Drying	
A	1	0.041	0.058	0.076	0.092	0.025	0.037	0.052	0.071	0.116
	2	0.030	0.044	0.061	0.077	0.021	0.030	0.043	0.060	0.083
B	1	0.018	0.025	0.033	0.040	0.010	0.016	0.021	0.028	0.069
	2	0.015	0.020	0.026	0.034	0.008	0.012	0.017	0.023	0.048

[1] Cement content, 6 sacks per cu yd; maximum size of aggregate, 1½ in.; specified slump, 4 in. maximum.

[2] Contraction of cement in mortar (Calif. 527-B): cement 1 (average of 4 job samples), 0.0547 percent; cement 2 (average of 4 job samples), 0.0373 percent.

[3] Concrete wet sieved through 1-in. sieve; average of 18.

[4] Average of 12.

[5] Curling of slabs on grade; measurements at time of maximum curling for season (40 days).

As the concrete was being placed in the decks, one 12-ft by 12-ft by 8-in. slab was cast on a cement-treated base and twelve 4- by 5- by 18-in. prisms and eighteen 3- by 3- by 11¼-in. prisms were molded from each aggregate-cement combination. Concrete placed in the smaller molds was wet sieved through a 1-in. sieve.

The prisms were moved to the laboratory at Sacramento on the day after molding. They were cured in fog at 73.4F to the age of 7 days. They were then measured for initial length and were placed in storage at 73.4F and 50 percent relative humidity. Changes in length were measured after 7, 14, 28, and 56 days of drying.

A small hole surrounded by a metal ring was cast near each corner and at the center of the slabs on grade. Metal rods were driven through the holes 4 ft into the subgrade, the upper half of the rod being protected from contact with the soil by a metal sleeve. Measurements of relative elevation were made with a dial gage supported on

TABLE 17

PREDICTION OF CONCRETE[1] SHRINKAGE[2] FROM MORTAR SHRINKAGE

Prism (in.)	Days of Drying	Aggt.	Cement	b[2]	Shrinkage (%)		Difference	
					Predicted[3]	Observed	Numerical	%
3 × 3 × 11¼	7	A	1	0.78	0.043	0.041	0.002	5
			2		0.029	0.030	0.001	3
		B	1	0.37	0.019	0.018	0.001	6
			2		0.014	0.015	0.001	7
	14	A	1	1.12	0.061	0.058	0.003	5
			2		0.042	0.044	0.002	5
		B	1	0.50	0.027	0.025	0.002	8
			2		0.019	0.020	0.001	5
	28	A	1	1.51	0.083	0.076	0.007	9
			2		0.056	0.061	0.005	8
		B	1	0.65	0.036	0.033	0.003	9
			2		0.024	0.026	0.002	8
	56	A	1	1.86	0.100	0.092	0.008	9
			2		0.070	0.077	0.007	9
		B	1	0.80	0.044	0.040	0.004	10
			2		0.030	0.034	0.004	12
4 × 5 × 18	7	A	1	0.51	0.028	0.025	0.003	12
			2		0.019	0.021	0.002	10
		B	1	0.20	0.011	0.010	0.001	10
			2		0.007	0.008	0.001	12
	14	A	1	0.74	0.040	0.037	0.003	8
			2		0.028	0.030	0.002	7
		B	1	0.30	0.016	0.016	0.000	0
			2		0.011	0.012	0.001	8
	28	A	1	1.04	0.057	0.052	0.005	10
			2		0.039	0.043	0.004	9
		B	1	0.42	0.023	0.021	0.002	10
			2		0.016	0.017	0.001	6
	56	A	1	1.43	0.078	0.071	0.007	10
			2		0.054	0.060	0.006	10
		B	1	0.56	0.030	0.028	0.002	7
			2		0.021	0.023	0.002	9

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[1] From Webber Creek bridges.

[2] In equation Y = bX.

[3] From mortar shrinkage X (Calif. 527B) using equation Y = bX.

the metal ring and with the stem in contact with the upper end of the rod. The difference between the average elevation of the four corners and the center of the slab was reported as "curl." Measurements were not corrected for warping due to temperature differential because it was not large and because each of the slabs was subjected to the same conditions.

Curling increased with time but leveled off at about 40 days, after which weather conditions caused a reduction in curling. There were no further increases up to the age of 100 days, the time of the most recent measurement.

Table 16 gives shrinkage results of the cements in mortar (Test Method No. Calif. 527B) and of the concrete prisms. Curl in the slabs on grade at the age of 40 days is also given in the table. The data of these tests may be used to determine agreement with certain findings presented earlier in the report.

Shrinkage of Cement in Concrete Related to That in Mortar.—For each of the cements there are two sizes of concrete test specimen, two aggregates, and four drying periods, making a total of 16 values for comparison with the cement mortar values. Predictions of concrete shrinkage Y from the mortar shrinkage X using Eq. 3, in which the value of b was selected for best fit, with differences from observed values given in Table 17. In most instances the predicted shrinkage differs from that observed by not more than 10 percent of the observed value. Two of the three cases in which the difference exceeds 10 percent are for 4- by 5- by 18-in. specimens dried for 7 days, in which the numerical values are very low and differences expressed as percentages are magnified. The results confirm those reported in Table 11 and discussed under "Analysis of Laboratory Studies."

Effect of Individual Factors on Cumulative Shrinkage.--The data of Table 16 have been used to calculate the shrinkage of the aggregate-cement combination A-1 from the remaining data. Table 18 gives the results of these calculations and compares them to the observed results. Separate calculations have been made under two assumptions:

1. The cumulative shrinkage is the sum of the individual factors; i.e.,

$$\text{A-1} = (\text{A-2} - \text{B-2}) + (\text{B-1} - \text{B-2}) + \text{B-2} \tag{5}$$

2. The cumulative shrinkage is the product of the ratios of the individual factors; i.e.,

$$\text{A-1} = \frac{\text{A-2}}{\text{B-2}} \times \frac{\text{B-1}}{\text{B-2}} \times \text{B-2} \tag{6}$$

TABLE 18

EFFECT OF INDIVIDUAL FACTORS ON CUMULATIVE SHRINKAGE[1]

Prism (in.)	Age (days)	Shrinkage[2] (%) Observed	Assumption 1	Assumption 2
3 x 3 x 11¼	7	0.041	0.033	0.036
	14	0.058	0.049	0.055
	28	0.076	0.068	0.075
	56	0.092	0.083	0.091
4 x 5 x 18	7	0.025	0.023	0.026
	14	0.037	0.034	0.040
	28	0.052	0.047	0.053
	56	0.071	0.065	0.073

[1] Concrete from Webber Creek bridges.

[2] Of aggregate-cement combination A-1 as observed and as calculated under two assumptions.

The data show that the shrinkage of combination A-1 is consistently underestimated when calculated under the first assumption, but is closely approximated under the second. This result confirms the finding that was developed from the data of Table 9 and discussed under "Combined Effect of Unfavorable Factors."

Slab Curling Related to Concrete Shrinkage.—It is obvious from Table 16 that the order of increasing slab curling is the same as that of shrinkage of the test specimens. Figure 12 shows the relationship of slab curling to shrinkage of 3- by 3- by 11¼-in. prisms after drying for 14 days and of 4- by 5- by 18-in. prisms after drying for 28 days. Considering that job-mixed concrete was used, the results are impressive in indicating that, under similar exposure, the amount of curling developed in pavement slabs is approximately proportional to the shrinkage of laboratory test specimens, also discussed in relation to the Topeka test road under "Pavement Performance Related to Shrinkage."

Warehouse Floor Performance

In 1924, Chapman (2) reported serious warping and cracking of a relatively new warehouse floor constructed above grade in two courses. The upper course was a 1:2 mortar of conglomerate screenings. Cracks divided the floor into rectangles 4 to 6 ft on a side which curled upward about 5/16 in. In general, the cracks were not visible on the underside of the floor; however, curling could be detected at junctions with brick-supporting walls. The author made laboratory shrinkage tests of 1:2 mortar using job and other sands, and also tests of concrete of job proportions. Shrinkage of the job mortar after 40 weeks of drying was about twice that of Ottawa sand mortar and about four times that of the job concrete. Approximately the same relationship resulted after short periods of drying. The evidence, therefore, is quite conclusive that job performance was in accordance with laboratory shrinkage results.

Effect of Reinforcement

Miller (31) attributes the dishing of thin slab floors, the sagging of shallow beams, and the drooping of marquees to warping deformations caused by nonsymmetrical position of the reinforcement. He found experimentally that, in nonsymmetrical sections, warping (curling) on drying is a function of the free shrinkage of the concrete. One means of reducing warping is to use concrete of low shrinkage.

Miller's findings may be extended to show a benefit from distributed reinforcement in concrete pavements. If the steel is placed near the upper surface, as is the usual practice, it can prevent or reduce the tendency of individual slabs to curl upward due to unequal drying. Profilograms obtained by California of the reinforced pavement of the Missouri test road (32) do not show the upward curling typical of nonreinforced pavements. This single illustration, however, may not constitute complete proof of the effectiveness of reinforcement in this respect because of unknown conditions at this site which may have greatly minimized curling. Even though it has been indicated that steel is effective in reducing moisture curling, it does not appear that it assists in reducing warping due to thermal differentials. The latter behavior may account for failure of reinforcement to improve performance in the ASSHO Road Test (33).

ANALYSIS AND SUMMARY

There is substantial evidence that suitably designed laboratory tests can be used to predict the effect of the characteristics of the constituents of concrete and the conditions of its manufacture on its shrinkage in service. The elapsed time to complete the tests need not be long. For most purposes, they can yield significant results in 7 to 21 days.

Shrinkage is dependent on the unit water content of concrete; however, the effect of materials having different characteristics cannot be foretold by a knowledge of their effect on water demand.

Factors such as aggregate size and gradation, slump, temperature of the concrete on discharge from the mixer, and the time of mixing or agitation have an effect on water requirement. These factors are subject to control within limits under the provisions of most specifications. Their combined effect, if adverse, is not likely to result in an increase in shrinkage of more than about 60 percent. The reduction in shrinkage that can be achieved by stringent control of these factors, though important, is much less than is possible through control of materials under specification requirements that are not presently in widespread use.

The amount of tolerable shrinkage depends on the design of each member and the function it is intended to perform. In many cases, there is considerable leeway in the amount of shrinkage that can develop before adverse effects are visually evident. On the other hand, it is reasonable to expect something less than the best in performance if shrinkage is allowed to increase too far beyond a practical limit. Combinations of unfavorable factors can result in an increase of severalfold in shrinkage. The probability of such an occurrence is augmented by the emphasis that is too frequently placed on high strength without proper consideration of other properties.

When the factor of safety against excessive shrinkage has been exhausted, experience shows that the cost of maintenance or repair is substantially greater than the cost of initial prevention.

The California Division of Highways has adopted the following requirements for the quality of materials as insurance against excessive shrinkage.

1. Portland cement is required to be Type II, low-alkali cement and the shrinkage developed in mortar shall not exceed 0.048 percent when tested by Test Method No. Calif. 527.
2. Aggregates are required to be sufficiently free from clay-like coatings or inclusions to meet test requirements for sand equivalent and cleanness value.
3. The maximum permissible dosage of chemical admixtures is limited to an amount that does not increase the shrinkage of concrete by more than 20 percent when tested by Test Method No. Calif. 530.

Although the restrictions were opposed at first by a few manufacturers, they are now accepted as a matter of course. In general, the manufacturers of portland cement and concrete aggregates have cooperated fully, once the reasons for the added requirements were fully understood. As far as known, there has been no increase in the price of cement due to the added requirements. There may have been a nominal increase in the cost of producing cleaner aggregates, but such added costs if any, have not been discernible in the bid prices for construction items involving portland cement concrete. There is some evidence that the cost of the completed work has been reduced.

In certain areas of the State, the only aggregates locally available are those having inherent characteristics that increase the shrinkage of concrete. To date, the California Division of Highways has not specified against their use. It would be desirable to eliminate aggregates of the higher inherent shrinkage-producing characteristics, particularly for use in nonreinforced pavements where curling is a major adverse factor affecting service. Nevertheless, the haul distances involved in importing other aggregates make it necessary to compare costs with benefits. Studies of possible economical means of improving aggregates with respect to their inherent contribution to shrinkage have not been encouraging to date. The adopted measures just described, together with previously adopted requirements for quality of materials and good construction procedures provide a significant margin of safety against excessive shrinkage. Further steps to effect a greater reduction in shrinkage logically should await additional studies as to costs involved and the benefits to be gained.

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Effect of Stress on the Dynamic Modulus of Concrete

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This paper presents the results of a preliminary investigation of the effect of stress intensity on the modulus of elasticity of concrete. Stress-strain diagrams are shown which were constructed for static tests and by using the vibratory data for transverse resonant vibrations at various stress intensities. The various tests were performed on the same cylinders, thereby using the same specimen for both the vibratory and static tests. The vibratory test is an adaptation of ASTM C215-60 with the specimens vibrated in their transverse mode.

The results of this investigation indicate that the dynamic modulus of elasticity increases as the intensity of stress on the specimen increases. The increase in the dynamic modulus of elasticity varied from 7 to 18 percent for different strength specimens, whereas the corresponding decrease in the static "tangent" modulus was 54 and 7 percent for the same specimens.

•IN A SENSE, only perfectly elastic materials have a modulus of elasticity. For many structural materials, the stress-strain relations are neither constant nor perfectly elastic. This is particularly true of concrete for which values of this ratio are generally understood to vary with magnitude and period of application of stress, with age and quality of concrete, and with moisture and temperature conditions. Furthermore, concrete is not perfectly elastic even at low stresses because it fails to resume completely its original dimensions on release of stress and continues slowly to deform over a long period of time under the action of sustained stress. Hence, insofar as concrete is concerned, the term modulus of elasticity is not a correct term because the stress-strain ratio includes plastic as well as elastic deformations.

The load deformation of a concrete specimen is dependent on two factors: the amount of stress and the length of time over which the stress is applied. The first type of deformation is due to the elastic properties of the concrete. The second type of deformation is the result of the inelastic or plastic properties of concrete.

In computing the total deformation of a concrete structure, the entire strain--plastic and elastic--must be used. If, however, the dynamic response of a concrete structure is desired, the modulus of elasticity, as found by the sonic or vibratory method, would govern the response of the structure and would be considerably different from that obtained in the static test.

It is difficult to define clearly all the reasons for the difference between the static and the dynamic moduli of elasticity, but the difference is probably in the varying amounts of plastic and elastic deformations that occur. Moreover, because of the stress-strain characteristics of concrete, it is probable that the modulus of elasticity of concrete, regardless of how it is determined, is a function of the stress intensity.

OBJECTIVES

Before undertaking a broad research study of the static, dynamic, and fatigue properties of concrete, an evaluation was made of the various experimental techniques used in concrete research. During this process, the effect of stress intensity on the dynamic modulus of elasticity of concrete was evaluated. The techniques used and results obtained for three representative specimens of different mixes and strengths are de-

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scribed. The methods used to study the elastic modulus of concrete used the same specimen for both the vibratory and the static tests.

TEST METHODS

Any method that is based on one specific property will have results that are dependent on the component in the mixture that has the greatest effect on the property measured. To obtain the modulus that governs the response of concrete structures, it is necessary to base the method on the action of the entire mass. Thus, the value determined will be the result of the nonhomogeneous mass acting singularly and not on any one component of the mass.

Static Method

This procedure, which measures the action of the entire mass and includes some plastic as well as the elastic deformation, is similar to the procedure covered in ASTM C469-61T, in which the load is applied slowly while the deformations are measured and the resulting stress-strain diagram obtained. The static method, even when the loads are applied at extremely high rates of speed, includes the plastic flow of the concrete in the measurement of strain.

Dynamic Method

The dynamic method used is similar to ASTM C215-60, in which the resonant frequency of the specimen is determined and by using the size and density of the specimen, the elastic constant is computed. Because of the extremely small deformations that occur as a result of the resonant vibrations, this method incorporates little, if any, plastic flow in the results. Moreover, because this method is based on the vibratory motion of the entire specimen, it should yield a modulus of elasticity that will govern the response of concrete either to vibratory and shock loadings or to static loads after the concrete has been subjected to sustained stresses. Therefore, it should determine what might be called the "purely elastic" modulus of elasticity of a specimen. The disadvantage of this method, however, is that the resonant frequency is determined at a zero stress and, therefore, the dynamic modulus of elasticity is the "purely elastic" property at a zero stress intensity. To overcome this disadvantage, the vibratory method was used with cylindrical specimens that were prestressed by post-tensioning bars along their longitudinal axis.

To reduce the irregularity of results often found in concrete testing, the static and dynamic procedures were performed on the same specimens.

Test Specimens

The specimens were 6 in. in diameter and approximately 30 in. long. The actual lengths were 30.27, 29.00, and 28.48 in. for specimens 1, 2, and 3, respectively. A 1¼-in. diameter hole was cast along the longitudinal axis of the cylinders. The purpose of the hole was to enable a prestressing rod to be inserted in the specimen. Each specimen was tested through the entire series of tests within a reasonable length of time, usually one or two hours. This was done to insure that the results would not be affected by a prolonged stress on the concrete.

The specimens were all cast of the same aggregate. The coarse aggregate was a subrounded material with a maximum size of ¾ in. Specimen 1 had a water-cement ratio of 0.684, and the mix by weight was 1, 2.92, 2.10. The average ultimate 28-day strength of this mix was 3,200 psi. Specimen 2 had a water-cement ratio of 0.60 and a mix by weight of 1, 2.18, 1.79. The average 28-day strength of this mix was 3,800 psi. Specimen 3 had a water-cement ratio of 0.546, and a mix by weight of 1, 2.05, 1.63. The average 28-day strength of this mix was 4,500 psi.

Static Equipment

The compressometer used to measure the longitudinal deformation of the specimen and thereby to determine the axial stress-strain curve was made of two 8-in. diameter cast steel yokes (Fig. 1). Each yoke was mounted on the specimen with three set screws, thus enabling each yoke to be entirely isolated and to form a plane perpendicular to the axis of the cylinder. The gage length of the compressometer was 8 in. This was determined by the two spacers that allowed the yokes to be 8 in. center to center. The unit deformation was found by averaging the total deformation as indicated on the two dial gages and then dividing by the gage length. The smallest unit deformation that could be read was approximately 3 micro-in. per in.

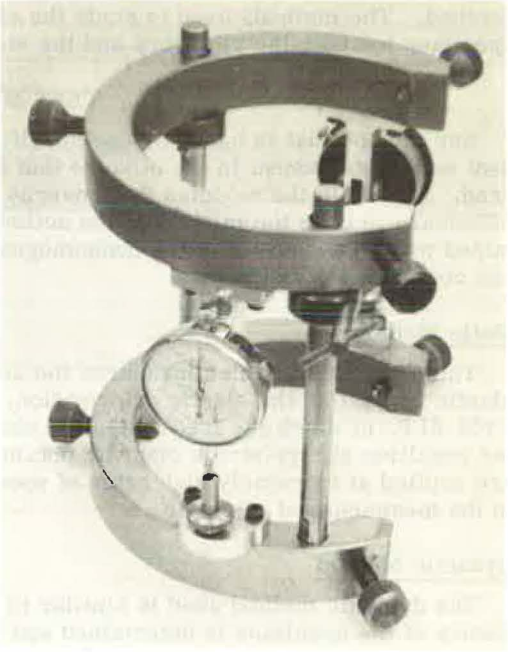

Figure 1. Compressometer.

Static Procedure

To determine the static stress-strain curve of the test specimens, a 200,000-lb Olsen testing machine was used. The load was applied at a rate of 0.05 in. per min. and readings were taken at approximately 5,000-lb increments.

Prestressing Procedure

The flexural resonance was determined for the prestressed specimens in order to compute the dynamic modulus of elasticity at elevated stresses. In applying the prestress, the specimen was first subjected to a compressive stress, then the $1\frac{1}{16}$-in. diameter prestressing rod was tightened. As the load was removed from the specimen, the rod absorbed the prestress. To determine the amount of prestress accurately, the prestress rod was instrumented with an SR-4 strain gage. This strain gage was calibrated after being mounted on the prestress rod to insure accuracy in the computation of the amount of the prestress. A specimen being prestressed in the Olsen testing machine is shown in Figure 2. The SR-4 strain indicator and switching box are shown with the dummy gage and the wires leading to the capping plate and thence to the strain gage mounted on the prestress rod. Concurrently, with the first prestressing of the specimen, a static stress-strain curve was determined.

Figure 2. Prestressing specimen.

Dynamic Equipment

The equipment used to determine the resonant frequency of the specimens consisted of an audio-oscillator, two amplifiers, an oscilloscope, a vibration pickup, and a driver. The equipment is shown in Figure 3 and corresponds to the equipment specified in ASTM C215-60.

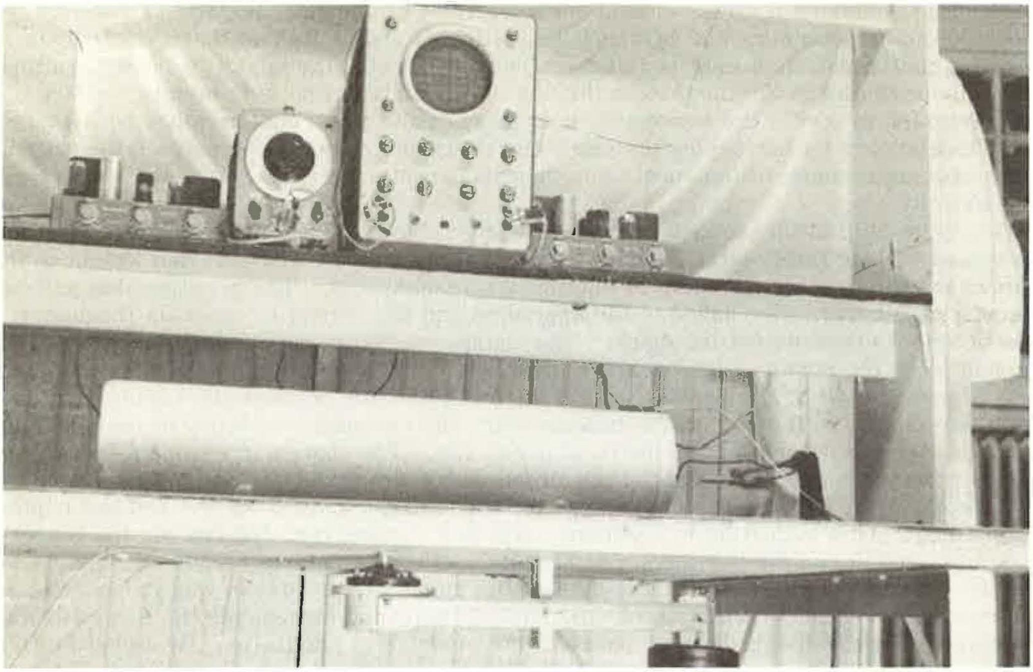

Figure 3. Vibratory modulus equipment.

Dynamic Procedure

The flexural or transverse mode of vibration was used in this investigation. The flexural mode of vibration was used to reduce the effect of the steel prestressing rod and capping plate. The steel prestressing rod and capping plates were needed to place the specimens in a stressed condition. The prestressing rod was mounted axially in the specimen so that there were no points of contact between the concrete specimen and the steel prestress rod. The weight of the rod, therefore, was carried by the capping plates which were in contact with the end of the concrete specimen. This was very carefully done to insure that the effect of the addition of the steel rod and capping plates could be considered the same as the addition of a mass at each end of a transversely vibrating beam.

Resonance in the specimen was indicated in one of two ways. The first method was that of noting the maximum vertical amplitude of the signal from the crystal pickup as applied to the vertical axis of the oscilloscope. This method makes use of the maximum amplitude of the specimen when vibrating at its resonance. The second method makes use of the frequency relationship between the driver and the specimen. When the specimen is in resonance, the driver frequency is exactly the same as the frequency of the specimen. By using a Lissajous figure, in which the driving frequency is entered on one axis and the specimen frequency is entered on the other axis, the frequency relationship can be easily determined. An oscilloscope was used in this capacity.

The specimen to be tested was mounted at its nodal points which are 0.22L from each end where L is the length of the specimen. In the case of the specimens that had been prestressed, the nodal points moved out toward the ends because of the added weight of the prestressing rod and the capping plates. Each time the specimen was thought to be in resonance, the pickup was moved along the specimen in such a manner as to show the mode of vibration at which the specimen was vibrating.

The method used in this study is based on the vibratory method of determining modulus of elasticity at zero stress. Using this method at various stress intensities, it is possible to construct a stress-strain curve entirely of dynamic moduli. To do this, the moduli are determined at different stress levels and plotted so that a continuous curve joins the points of tangency at the stresses at which the moduli were determined. The only difficulty in this method is the use of an additional outside member which must be used to stress the concrete. It is this outside or foreign member which may introduce errors by increasing the mass end fixity and possibly the potential energy in the restoring moment of the specimen, thus incorrectly changing the dynamic modulus of elasticity.

The first step in this testing procedure was to obtain the resonant frequency of the specimen without the prestress rod. By using this value and the size and weight of the specimen, the dynamic modulus of elasticity was computed. The prestressing rod and capping plates were then added to the specimen and the change in resonant frequency was noted. In this step of the testing, the change was often very irregular. This was thought to be the result of the prestress rod being only hand-tight on the specimen, and thus allowing additional vibrations to occur in the junction between the capping plates and the washers on the rod. To eliminate this, the resonant frequency of the specimen with the rod was determined at the first prestress. The change in resonant frequency between the plain specimen and the specimen with the rod and capping plates prestressed to approximately 400 psi was considered to be due to the addition of the rod and capping plates only. This reduction in resonant frequency varied from 228 cps for the longer specimen to 350 cps for the shorter specimen. After the natural frequency was obtained for the specimens with the first prestress, the amount of prestress was increased and the resonance again determined. If the natural frequency increased, the increase was assumed to be the result of an increase in the modulus of elasticity. By using the first prestress resonance as the base resonance for the specimen with the rod and plates, it was assumed that the modulus of elasticity for the base resonance or first prestress was equal to the dynamic modulus of elasticity of the specimen at zero stress. The additional moduli were computed by noting that the modulus of elasticity of a particular

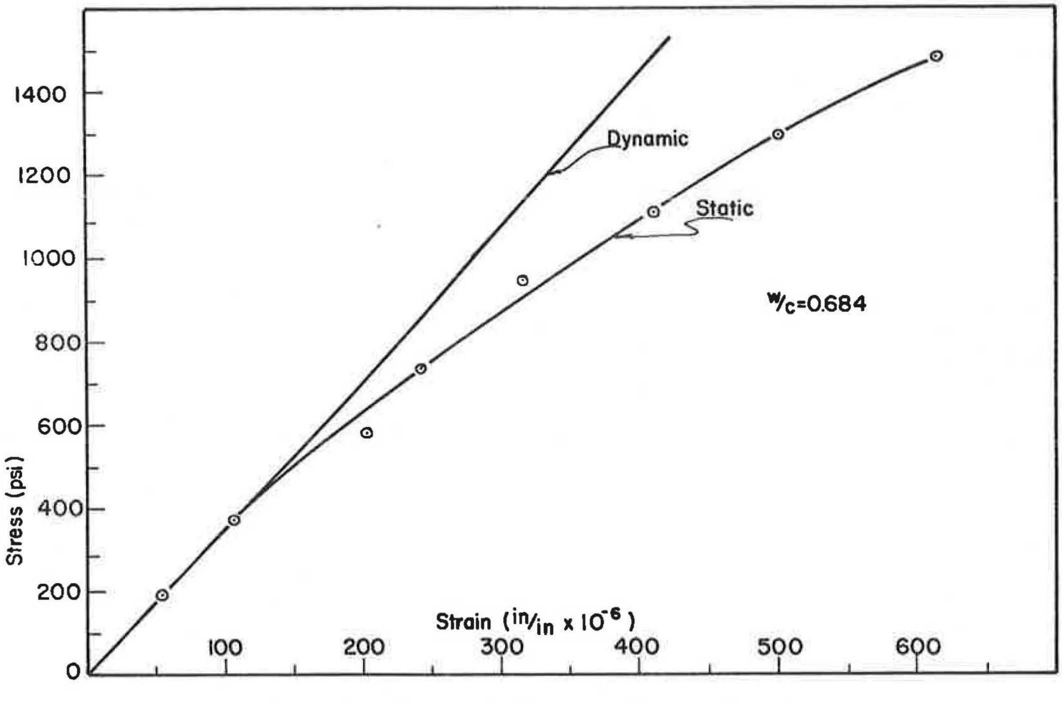

Figure 4. Stress-strain curve for specimen 1.

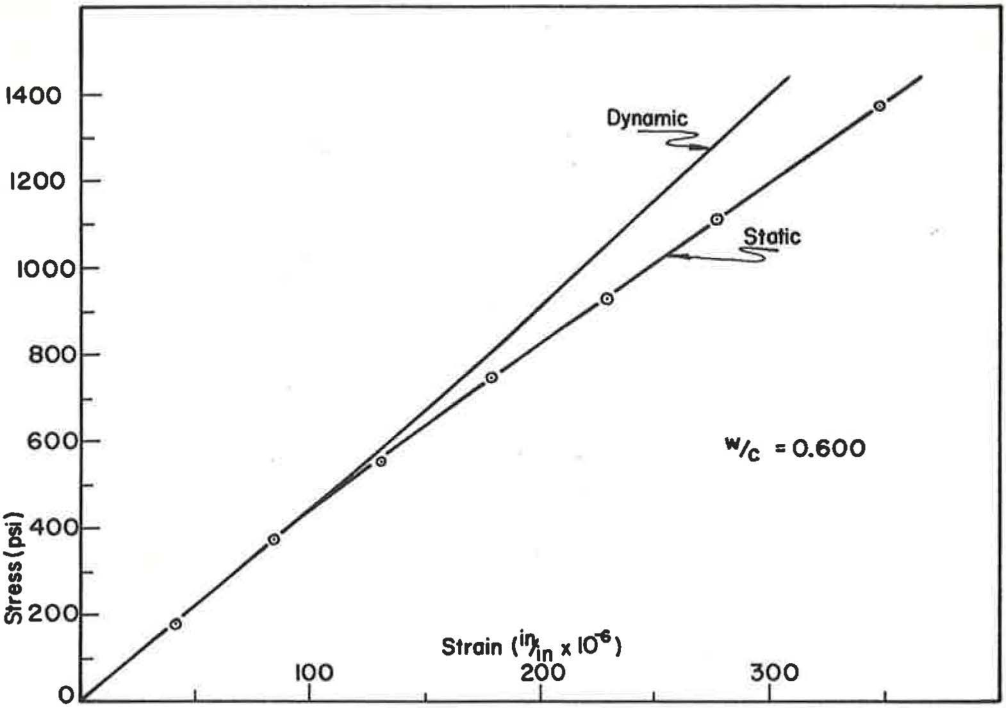

Figure 5. Stress-strain curve for specimen 2.

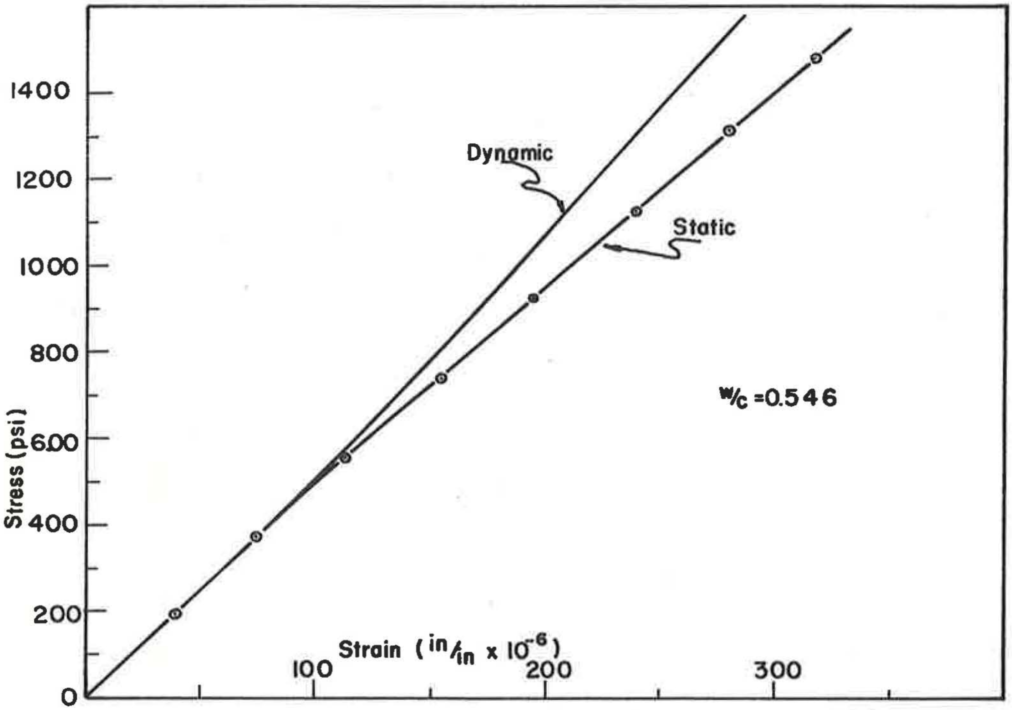

Figure 6. Stress-strain curve for specimen 3.

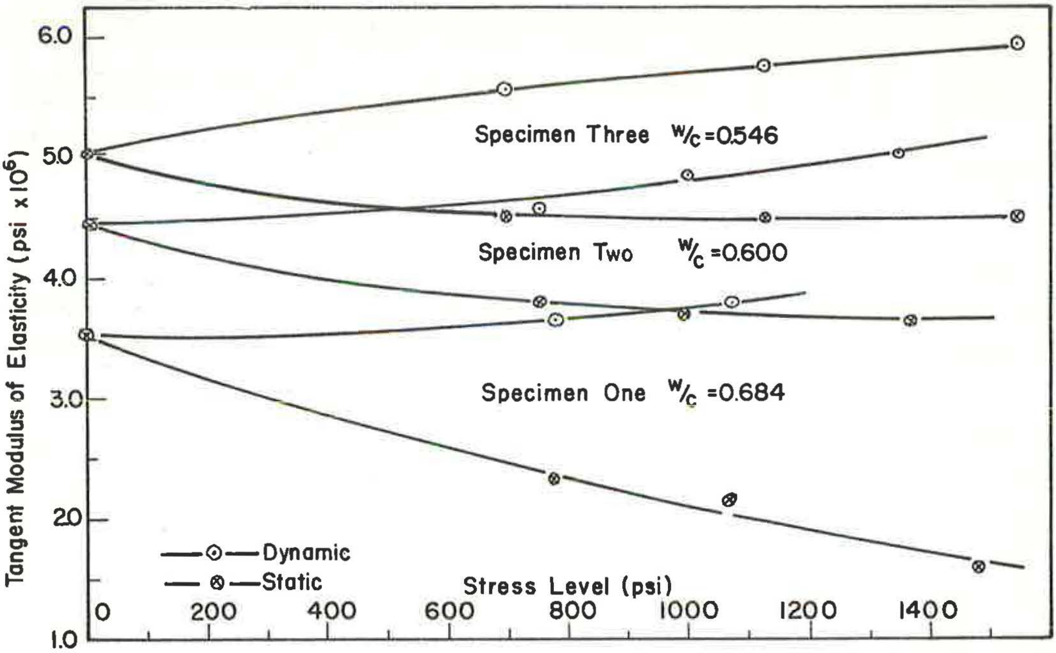

Figure 7. Variation in tangent modulus of elasticity with stress level.

specimen is directly proportional to the square of the resonant frequency. The dynamic modulus of elasticity was then determined for each specimen at each prestress by multiplying the square of the ratio of the stressed natural frequency to the base natural frequency by the unstressed dynamic modulus of elasticity. Because the maximum change in stress during the vibration is very small, the values of the moduli from which the results are based are values obtained at very small increments of stress. Therefore, the results should not include any appreciable amount of plastic flow. The stress-strain curves as obtained by the procedures previously discussed for the static and dynamic tests are shown in Figures 4, 5, and 6 for specimens 1, 2, and 3, respectively.

RESULTS

It is evident from Figures 4, 5, and 6, that the dynamic modulus at zero stress agrees closely with the initial tangent modulus of elasticity for the static results. Moreover, both the static initial tangent modulus and the dynamic modulus at zero stress increase for the higher strength specimens. The static compression test results show the typical stress-strain curve which is concave downward. The modulus of elasticity for this curve decreases with increasing stress. However, the dynamic moduli increase for an increasing stress and form a stress-strain curve that is uniquely concave upward. This is not unreasonable, however, if the Watertown Arsenal Report of 1899 and other studies (1) of natural building stones are consulted. These studies indicate that natural stone has a concave upward stress-strain curve. Inasmuch as concrete might be considered a "green" form of natural building stone, it is evident that by eliminating creep it might be expected to act as a natural stone. Moreover, the results presented herein agree with and substantiate the results obtained by Obert in 1941 with a different method (2).

The area between the dynamic curve and the static curve represents what might be considered "creep strain," which decreased for the higher strength specimens.

The best indication of the difference between the static and dynamic results can be represented by plotting the dynamic moduli at elevated stresses and the static tangent moduli at each similar stress against the stress magnitude, as shown in Figure 7. The increase in the dynamic modulus of elasticity was 7.4, 13.2, and 18.3 percent, and the decrease in the static tangent modulus was 54.8, 16.3, and 6.96 percent for specimens 1, 2, and 3, respectively. Obert found an increase in the dynamic modulus of 8.2 to 13.0 percent of the initial tangent modulus of elasticity (2), which is in the range obtained in this research.

CONCLUSIONS

The results of the tests performed in this investigation show that the modulus of elasticity of concrete has a tendency to increase as the stress increases, provided the inelastic creep can be eliminated. In this study, the creep was eliminated or preactivated by prestressing the specimens and by the use of a very small superimposed stress during the determination of the elastic modulus by the vibratory method.

The difference between the static modulus and the dynamic modulus was less for the higher strength specimens, which indicates the possible application of this method in studying the elastic properties used for deflection calculations in high strength concrete. The results also suggest the use of this method in studying the repeated load response of concrete.

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Discussion

ERNEST E. MC COY, Chief, Thermal Research and Field Durability Section, Concrete Division, U. S. Army Engineer Waterways Experiment Station, Jackson, Mississippi—The author is to be congratulated for his excellent paper on a subject that is currently of increasing interest and importance. In 1939 Obert (5) described results of vibration tests of concrete beam specimens under stress; he used the simply supported (hinged-ends) mode of vibration. After the author's paper was presented, a single concrete specimen was tested at the writer's laboratory to investigate whether it is feasible to vibrate a specimen under longitudinal stress (compression), in the fixed-ends mode of vibration.

The specimen was a 6- by 12-in. cylinder. Its age was 8 months, weight 28.24 lb, air content 1.8 percent, and dynamic modulus 3.30×10^6 psi. The specimen was from a set having approximately 2,400-psi compressive strength at 28 days age, at which age it had been subjected to a hydraulic pressure of 4,000 psi in a test for air content. It had then dried in laboratory air for several months and its surface presumably was covered with microscopic shrinkage cracks at the time of the tests described below.

The specimen was subjected to two vibration tests with incremental compressive stress. For the first test, load was applied in five 100-psi increments and a final 500-

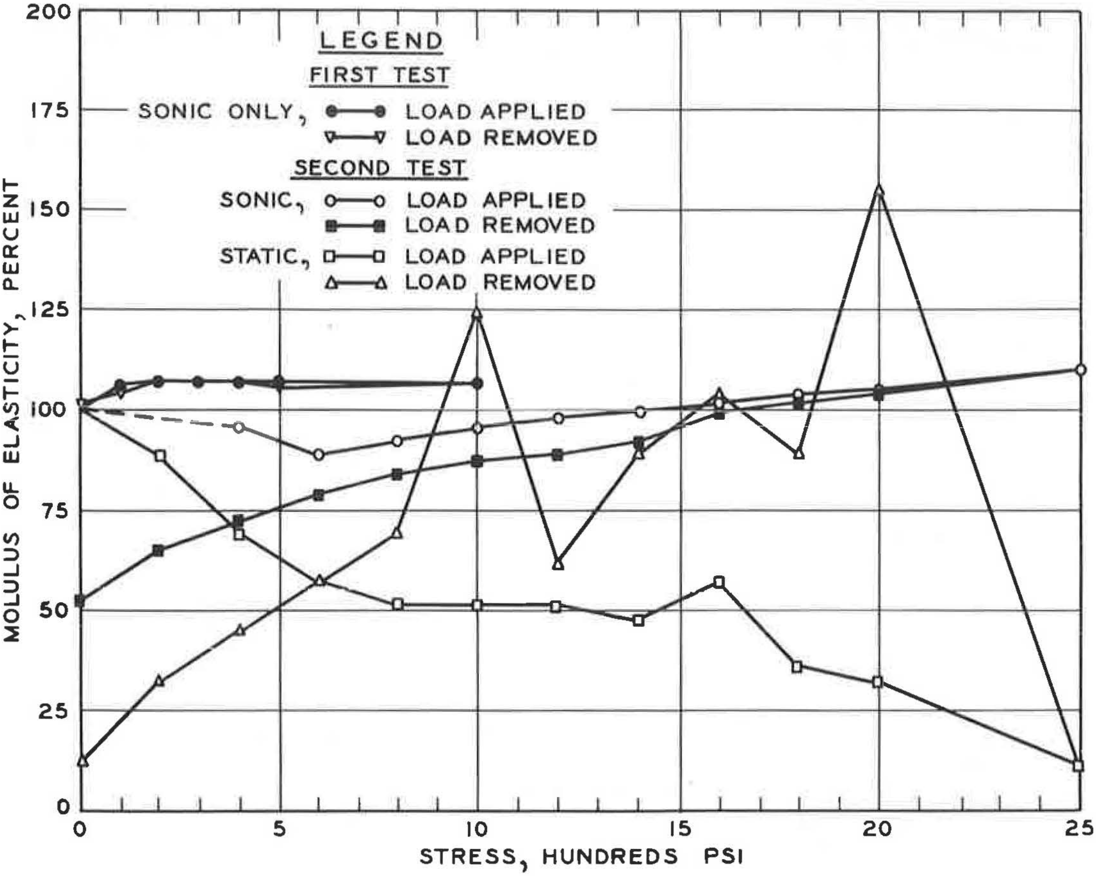

Figure 8. Variations of dynamic and static modulus with stress.

psi increment to 1,000 psi in a universal testing machine, and load was removed by the same increments. The resonant frequency was recorded for each increment. After six days the specimen was capped with a very thin layer of high-strength plaster, and four strain gages of 2½-in. gage length (wired to read as one gage) were mounted at 90° intervals. The next day the specimen was tested to 2,500 psi by increments of 200 to 2,000 psi and a final increment of 500 psi. Load was removed by the same increments. The rate of loading was 175 psi per min for each test. The initial frequency for the second test was obtained with the specimen properly supported for the free-free mode, and after the loading test was completed, the frequency was checked in the free-free mode, agreeing to 1 percent with the final frequency obtained for the loading test.

Figures 8 and 9 show the results. All frequencies on which (when squared) changes of modulus were based were assumed to be for vibration in the fixed-ends mode. According to simple formulas (2), there should be no difference between free-free and fixed-ends resonant frequencies.

The frequency at each point for the unloading cycle of the first test agreed to 1 percent with the loading cycle frequency. All the frequencies for this test, however, were about 5 percent lower than the predetermined free-free frequency. The pickup was taped to the specimen for the free-free test and was not removed for the incremental test; however, for the latter the pressure of the driver against the specimen may have been slightly higher.

It was thought that capping the specimen would improve free-free and fixed-ends frequency agreement for the second test. As mentioned, the initial frequency for the

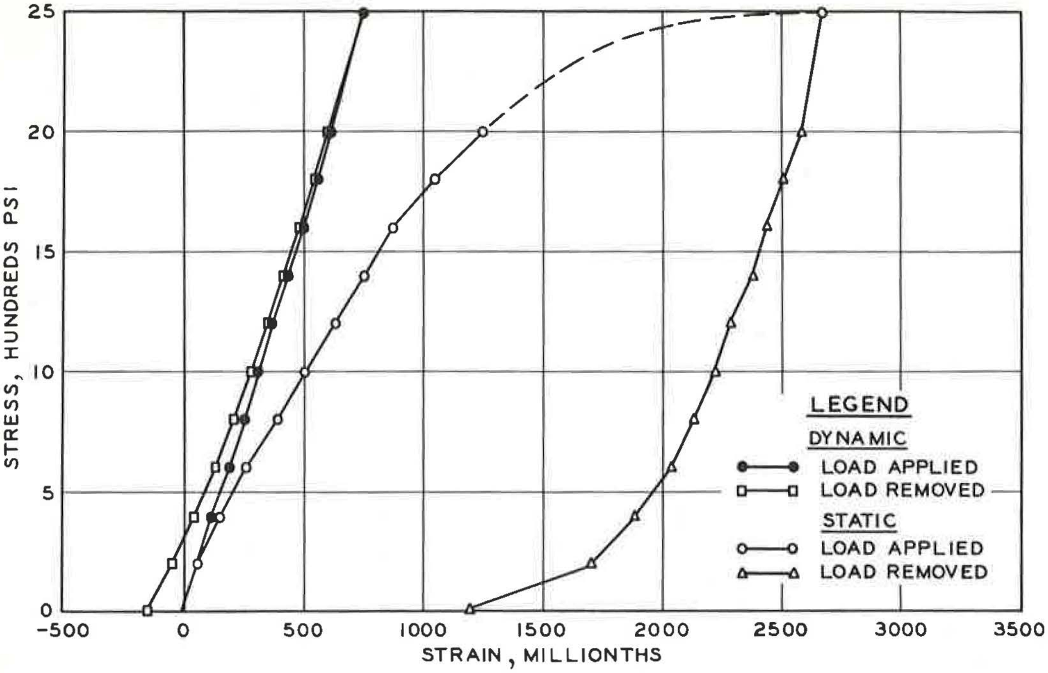

Figure 9. Stress-strain diagram, second test.

second test was obtained with certainty for the free-free mode. At zero load and at 200 psi, however, it appeared that the only frequencies obtainable were not relevant and they were not used. On removal of load, the squared frequencies, including the zero-load frequency, followed the smooth curve shown. The dip in the loading cycle curve can hardly be understood without further testing and study. A very subdued tendency for the vibration to be influenced by the simply supported mode, which has a lower frequency, may have affected the frequency for this test, and also for the first test. Beginning at 600 psi, the dynamic modulus curve closely resembles that for specimen 3 of the author's investigation, increasing 21 percent as compared with 18.3 percent. The increase of dynamic modulus beyond about 2,000 psi is noteworthy because the specimen had evidently been loaded critically beyond the yield point, as shown by strain measurements.

The zero static modulus (3.2×10^6 psi) for the strain gage measurement was obtained by extrapolation of the first three points. The percentage changes (Fig. 8) are for the chord modulus based on the measured stress and strain increments between consecutive points. The plateau between about 600 and 1,600 psi is considered to be evidence of the closing of microscopic cracks before critical yielding. A very considerable amount of creep or yield was observed to be in progress above 2,000 psi.

The results of the second test show that there was a very considerable divergence between static and dynamic modulus for the loading cycle, as did the results of the tests reported by the author and by Obert. The method employed by the author does not permit rapid simultaneous testing for both the static and dynamic modulus; that employed by Obert would do so with the addition of strain gages. Both methods require the use of end plates for which satisfactory corrections can be applied.

For the stress-strain diagram (Fig. 9), the apparent strains for the second dynamic test were computed by the method used by the author. The negative strains for a portion of the unloading curve are without special significance, merely reflecting the change in sonic modulus to a considerably reduced value as a result of overloading. The loading parts of the curves in Figure 9 are quite similar to the curves shown in Figures 4, 5, and 6.

The results of these tests provide evidence of the possible usefulness of the fixed-ends mode of vibration. The advantage of the method, if it can be perfected, would be continuous rapid testing of a relatively unencumbered specimen. It appears, however, that for further investigation of vibration in this mode the specimen should be firmly fixed to the testing machine platens or to relatively large and heavy auxiliary plates. It is contemplated that high-strength plaster or epoxy will be used for this purpose in an additional test. Apparently the amplitude of the vibrations at the ends is so small (only a few microinches) that imperfect contact, especially at low load, affects the test.

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D. A. LINGER, Closure—Mr. McCoy's discussion presents several important aspects and adds to the understanding of the dynamic modulus characteristic of concrete. The research of Obert previously mentioned is significant because in it are the basic ideas enlarged on by Mr. McCoy and the author. However, the limitations of the various methods used in determining the dynamic modulus at elevated stresses are noteworthy. Obert's method necessitated corrections amounting to 25 percent of the total change in frequency. These corrections were needed to account for the mass of the capping plates and the change in the vibrating condition. Hence, the correction applied by Obert was larger than the increase in the dynamic modulus obtained. Other problems exist inasmuch as it has been shown by Timoshenko (7) that an axial load changes the vibrating system and hence changes the natural frequency of vibration. The effect of an axial compressive load on the vibration of a hinged-end beam or a free-free beam results in a decrease in the frequency of the system by an amount equal to $\sqrt{1 - P/P_c}$ in which P is the axial load, and P_c is the Euler buckling load. If this is the only effect of the axial load, it is found that the reduction in frequency for a 6- by 12-in. cylinder is less than 0.1 percent, and for a 6- by 30-in. specimen is only approximately 0.1 percent. Therefore, the effect of the axial load on the hinged-end or a free-free vibrating system is negligibly small, if the end conditions and the fundamental mode of vibration are maintained.

The effect of changes in mode or type of vibration can, however, be quite large. The fixed end condition necessary for the fixed-fixed mode of vibration is extremely difficult to obtain, and Mr. McCoy is to be congratulated on the close agreement of the fixed-fixed vibration frequency of the second loading with the anticipated value. Vibration tests similar to Mr. McCoy's have been conducted by the author and it was found that the capping plates, type of testing machine, and the amount of axial load affected the end fixity of the cylinder and hence, the mode of vibration. This can be exemplified theoretically by noting that the percentage reduction in natural frequency of a beam from the fixed-fixed condition to the hinged-end condition is 57 percent. During a study of the elastic properties of the beam specimen, this change (or any small part of it) would be included in the calculations for the change in the dynamic modulus. Therefore, it is apparent that the type of test used to evaluate the dynamic modulus of materials needs close and careful study.

This brief discussion of the methods used should not detract from the results obtained. It is important to point out that the "unknown" factors just mentioned have a tendency to reduce the natural frequency or the resulting modulus of elasticity. From this it would seem reasonable that the increase in the dynamic modulus might have been even larger if the other "unknown" factors could have been held constant. Moreover, the increase that does occur in the dynamic modulus is extremely important in the dynamic analysis of concrete structures. During a recent comprehensive evaluation of the dynamics of highway bridges, it was found that the modulus of elasticity of the structural concrete needed to be considerably larger in order to obtain any agreement between the theoretical natural frequency of the bridges and the experimentally obtained actual natural frequency. In this case any theoretical dynamic analysis of the structures would have included considerable error if the static value of the modulus of elasticity was used in the computations.

The author is indebted to Mr. McCoy for his discussion, and would like to again point out the need for additional research on this subject in an era in which dynamic or blast loadings are becoming important concepts in design.

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