

## Tarun R. Naik, editor



# TEMPERATURE EFFECTS ON CONCRETE

A symposium sponsored by ASTM Committee C-9 on Concrete and Concrete Aggregates Kansas City, MO, 21 June 1983

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## Foreword

The symposium on Temperature Effects on Concrete was held in Kansas City, Missouri, on 21 June 1983. The event was sponsored by ASTM Committee C-9 on Concrete and Concrete Aggregates. Tarun R. Naik, of the University of Wisconsin at Milwaukee, presided as chairman of the symposium and also served as editor of this publication.

## Related ASTM Publications

Cement Standards--Evolution and Trends, STP 663 (1979), 04-663000-07

Significance of Tests and Properties of Concrete and Concrete-Making Materials, STP 169B (1978), 04-169020-07

Fineness of Cement, STP 473 (1970), 04-473000-07

Cement, Concrete, and Aggregates, ASTM journal

## A Note of Appreciation to Reviewers

The quality of the papers that appear in this publication reflects not only the obvious efforts of the authors but also the unheralded, though essential, work of the reviewers. On behalf of ASTM we acknowledge with appreciation their dedication to high professional standards and their sacrifice of time and effort.

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### Introduction

A symposium on Temperature Effects on Concrete, sponsored by ASTM Committee C-9 on Concrete and Concrete Aggregates, was held in June 1983 at Kansas City, Missouri. This volume contains ten papers, eight of which were presented at that symposium. The authors come from a variety of geographical areas, including the United States, Canada, and England.

The international aspect of this volume is reflected in the papers. The temperature effects on concrete described herein take place under conditions that vary from Arctic environments to high-temperature exposures of 600°C. While some of the authors have also presented findings of investigations for more general use—namely, the usual cold and hot weather conditions—one paper has even presented test results of concrete subjected to cryogenic temperatures.

The editor hopes and anticipates that this book will be of benefit to many engineers and researchers interested in temperature effects on concrete. Also, the references at the ends of the individual papers will be of benefit to readers seeking additional information for detailed study of the subject of temperature effects on concrete.

The editor would like to take this opportunity to express his appreciation to the reviewers of these papers for their timely reviews. He is also sincerely grateful to Dr. Vance Dodson and Herman Protz, members of ASTM Committee C-9 and Subcommittee C09.02 on Research, for their help in organizing the symposium. The continuous and prompt help provided by the publications department of ASTM is also very much appreciated.

#### Tarun R. Naik

University of Wisconsin at Milwaukee, Milwaukee, WI 53201; symposium chairman and editor.

### Strength Development of Concrete Cured Under Arctic Sea Conditions

**REFERENCE:** Aitcin, P.-C., Cheung, M. S., and Shah, V. K., "Strength Development of Concrete Cured Under Arctic Sea Conditions," *Temperature Effects on Concrete, ASTM STP 858*, T. R. Naik, Ed., American Society for Testing and Materials, Philadelphia, 1985, pp. 3-20.

**ABSTRACT:** Two sets of experiments simulating the curing conditions of concrete caisson constructions in the Arctic were carried out at Sherbrooke University, Province of Quebec, Canada, and at Nanisivik, in the extreme north of Baffin Island, Canada (73° north). More than 500 concrete specimens were tested for various ages and initial curing periods. After they were cast, the concrete specimens were initially cured at about 4°C (39°F) for 3 to 15 h and then immersed in seawater at 0°C (32°F) until testing. Their compressive strengths at different ages, up to one year, and Young's modulus at 28 days were compared with those of specimens of the same concrete and same age cured under room temperature.

These two sets of experiments have shown that if 9 h of initial curing at about  $4^{\circ}C$  ( $39^{\circ}F$ ) is allowed for the concrete before immersion in seawater at  $0^{\circ}C$  ( $32^{\circ}F$ ), the design compressive strength of the concrete can be achieved at 56 days. The rate of development of compressive strength during the first two weeks is slow because of the low temperature of the curing environment.

The temperature of the fresh concrete and its water/cement ratio are the two most important parameters that determine the early strength of the concrete.

**KEY WORDS:** low-temperature curing, Arctic Sea, concrete caisson construction, compressive strength, Young's modulus, concrete

Engineers have been successfully using concrete in all kinds of environments. When correctly designed and proportioned for its environment, hardened concrete can last for many years. In fact, very often the most critical period in the life of concrete is when it changes from the freshly mixed state to a hardened solid. During this time, an excess of water exists in the paste; its freezing or too-rapid drying can cause permanent damage and may lead to premature ruin of the concrete structure.

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As more development takes place in the Arctic Sea because of the discovery of rich mineral deposits and promising oil and gas fields, more concrete will be used in this area. The average summer temperature in this region is only about 4°C (39°F), and the seawater has an average summer temperature of between -1°C and 0°C (30 to 32°F).

Presently, most of the concrete marine structures erected in the Arctic are cast in the south and then towed north under very difficult conditions, such as the presence of icebergs, frequent fogs, and a very short navigation period. If the site conditions permit, it would be advantageous to build these structures close to the installation site. This may result in great savings in transportation, installation costs, and construction time.

The purpose of this paper is to report an investigation of the possibilities of casting, curing, and obtaining good-quality concrete in the cold Arctic Sea water environment. The paper also reports the simulation and study of the effects of current caisson construction practice, with which the concrete could be exposed to cold seawater within 3 to 10 h after placement.

An intensive literature survey has shown that there is insufficient information about the effects on concrete strength and durability resulting from such a construction practice. Therefore, Public Works Canada initiated two experimental studies simulating the curing conditions of concrete used in slip-formtype caisson construction in the Arctic Sea.

#### **First Simulation**

The first simulation was made at Sherbrooke University, Quebec, Canada, with three ready-mix concrete mixes with water cement ratios by weight of 0.45, 0.50, and 0.55. The compositions of these concretes are given in Table 1. A total of 150 cylindrical specimens, 150 by 300 mm (6 by 12 in.), and 4 beams, 150 by 150 by 900 mm (6 by 6 by 36 in.), were cast from each concrete. After casting, these specimens were immediately placed in a cold storage room at  $4^{\circ}C$  (39°F) (Fig. 1). After 3 h of initial curing, 8 series of 3 specimens each were immersed in 200-L barrels (55 U.S. gal) of artificial seawater placed in a second cold storage room at  $0^{\circ}C$  (32°F) (Fig. 2). After 6, 9, 12, and 15 h of curing at  $+4^{\circ}C$  (39°F), 32 more series of 3 specimens each were immersed in the same manner in the seawater (Table 2). The seawater was reconstituted by dissolving 35 g of commercial deicing salt in every litre of fresh water (0.29 lb/gal U.S.). The chemical composition of the salt is given in Table 3. A reference series cured at  $20^{\circ}C$  (68°F) in lime-saturated water was also taken.

All the specimens were unmolded 24 h after their casting and replaced in the seawater at 0°C ( $32^{\circ}F$ ). At one day plus 3 h, the first set of 3 specimens in seawater was tested in compression as well as 3 reference specimens cured at 20°C ( $68^{\circ}F$ ). Three specimens of each concrete were later tested at one day plus 6 h, one day plus 9 h, one day plus 12 h, and one day plus 15 h, according to their sequence of introduction in the seawater at 0°C ( $32^{\circ}F$ ).

		W/C Ratio	
	0.45	0.50	0.55
Water, kg/m <sup>3</sup>	165	165	165
Cement, kg/m <sup>3</sup>	370	340	300
Coarse aggregate, kg/m <sup>3</sup>	1020	1035	1000
Fine aggregate, kg/m <sup>3</sup>	810	825	895
Slump, mm	55	45	50
Air content, %	4.5	4.0	4.0
Temperature, °C	16	4	9

TABLE 1—Composition and properties of the three concrete mixes used in the first simulation.<sup>a</sup>

"To obtain the concrete composition in pounds per cubic yard, multiply the numbers of kilograms per cubic metre by 1.68.



FIG. 1-Concrete specimens in the initial curing period at 4°C (39°F).

Compression tests were performed at 7, 14, 28, 56, and 91 days, six months, and one year, according to the ASTM Test for Compressive Strength of Cylindrical Concrete Specimens (C 39-83a). Modulus of rupture and Young's modulus tests were also performed at 14 and 28 days, according to the ASTM Test for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293-79) and the ASTM Test for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression (C 469-81) standards, respectively.

The three concrete mixes were delivered by a local ready-mix producer between 26 Jan. and 4 Feb. 1982, when the outside temperature varied between

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FIG. 2—Curing of the concrete specimens in reconstituted seawater kept at  $0^{\circ}C$  (32°F).

Initial	Number of Specimens Tested at Various Testing Ages						Total		
Period at 4°C	1 Day	7 Days	14 Days	28 Days	56 Days	3 Months	6 Months	1 Year	Specimens
3 h	3	3	3	3	3	3	3	3	24
6 h	3	3	3	3	3	3	3	3	24
9 h	3	3	3	3	3	3	3	3	24
12 h	3	3	3	3	3	3	3	3	24
15 h Bafaran as	3	3	3	3	3	3	3	3	24
specimens	3	3	3	3	3	3	3	3	24
modulus			2	2	•••		•••		4

TABLE 2-Testing program of the first simulation (cylinder size: 150 by 300 mm).

TABLE 3—Chemical composition of the salt.

	Na	Ca	Mg	К	Cl	H <sub>2</sub> O	Nonsoluble	Total
Percent by weight	37.5	0.5	0.02	0.02	58.5	58.5	1.1	97.7

 $-20^{\circ}C$  ( $-4^{\circ}F$ ) and  $-4^{\circ}C$  ( $25^{\circ}F$ ). During this period it was very difficult to control the delivery temperature of the concrete. The delivery temperatures for water/cement (W/C) ratios of 0.45, 0.50, and 0.55 were  $16^{\circ}C$  ( $61^{\circ}F$ ) (hot water, heated aggregates),  $4^{\circ}C$  ( $39^{\circ}F$ ) (cold water, heated sand), and  $9^{\circ}C$  ( $48^{\circ}F$ ) (hot water, cold aggregates), respectively.

The temperature of the concrete specimens was frequently measured using thermocouples until the last cylinders were immersed in the artificial seawater. The temperature of the specimens in the seawater was also measured during the first 15 h (Fig. 3).

#### **Compressive Strength Results**

The average compressive strength results are shown in Tables 4, 5, and 6 in megapascals and in percentages of the 28 days' compressive strength in Figs. 4, 5, and 6.

The following observations may be made from these figures and tables.

1. After 24 h of curing in the seawater at  $0^{\circ}C$  (32°F) the compressive strength of the concrete specimens was always lower than that of the specimens cured



FIG. 3—Temperature measurements of the concrete specimens.

Initial Cooring			A	ge of Te	sting, day	s		
Period at 4°C	1	7	14	28	56	91	180	365
3 h	2.8	26.4	35.2	37.9	31.3 <sup>a</sup>	44.9	41.8	49.3
6 h	6.2	30.7	40.4	43.6	50.7	51.2	55.5	57.8
9 h	7.3	31.7	40.7	46.1	50.8	53.2	54.2	58.8
12 h	9.0	31.4	40.3	44.4	53.5	53.4	57.7	57.6
15 h	11.3	32.8	38.9	43.0	53.2	50.3	58.3	59.2
Reference								
specimens	20.0	29.7	35.1	37.0	44.8	45.3	47.4	53.

TABLE 4—Compressive strength results, MPa (average of three specimens), for concrete specimens with a 0.45 W/C ratio.

<sup>a</sup>Broken specimen.

 TABLE 5—Compressive strength results, MPa (average of three specimens), for concrete specimens with a 0.50 W/C ratio.

			A	ge of Te	sting, day	ys.		
Period at 4°C	1	7	14	28	56	91	180	365
3 h	1.1	19.7	23.5	31.1	28.5	36.0	42.0	44.7
6 h	1.9	20.9	26.0	33.3	34.8	39.2	40.4	
9 h	3.5	22.1	27.7	35.7	38.8	44.5	48.7	47.5
12 h	5.5	24.9	29.5	36.2	39.9	44.0	47.9	49.0
15 h	6.6	25.0	29.7	36.5	41.6	46.6	47.1	49.3
Reference								
specimens	12.8	27.8	31.2	36.0	39.0	41.6	43.5	48.6

under normal conditions. The lowest strengths were observed with the coldest concrete  $[W/C = 0.50; 4^{\circ}C (39^{\circ}F)]$ , which had the shortest initial curing period at 4°C (39°F) (3 h). The highest strengths were observed with the richer and hotter concrete  $[W/C = 0.45; 16^{\circ}C (61^{\circ}F)]$ , cured for the longest period of time at 4°C (39°F) (15 h).

2. At 7 days for the richest mix (W/C = 0.45), all the specimens cured in the seawater had a higher compressive strength than the normally cured ones, except for those that had been kept only 3 h at 4°C (39°F). For the two other mixes, the compressive strength of the specimens cured in the seawater was lower than that of the concrete specimens cured under normal conditions.

3. At 14 days, the compressive strength of the specimens of the two weaker mixes of concrete (W/C = 0.50 and 0.55) cured in seawater was almost equal to the compressive strength of the reference cylinders. The longer the exposure at 4°C (39°F), the stronger the concrete.

4. At 28 days, all the specimens cured in seawater with an initial 9-h curing period at  $4^{\circ}C$  (39°F) were stronger than the reference specimens. A 25% in-

			A	ge of Te	sting, day	ys		
Period at 4°C	1	7	14	28	56	91	180	365
3 h	2.1	15.6	27.1	31.8	33.7	36.3	39.8	38.7
6 h	3.4	20.7	29.5	33.8	34.5	37.6	42.3	
9 h	5.0	24.3	31.9	37.0	40.7	41.2	47.6	47.0
12 h	6.1	24.4	31.8	36.6	41.4	44.8	47.6	48.0
15 h	7.2	25.0	32.2	38.1	40.8	45.4	47.0	46.8
Reference specimens	12.6	27.8	30.0	35.1	39.2	41.4	41.8	45.2

 TABLE 6—Compressive strength results, MPa (average of three specimens), for concrete specimens with a 0.55 W/C ratio.



FIG. 4—Influence of the curing conditions on the compressive strength at different ages.



FIG. 5-Influence of the curing conditions on the compressive strength at different ages.

crease in the compressive strength was observed for the 0.45 W/C ratio concrete that had been cured 9 h at  $+4^{\circ}C$  (39°F).

5. At 56 days, 91 days, six months, and one year, similar results have been observed. Moreover, at one year the compressive strength of all the specimens cured in the seawater at  $0^{\circ}$ C (32°F) was higher than the 28-day compressive strength of the reference specimens cured at room temperature.

#### Young's Modulus Measurements

Young's modulus values are presented in Table 7. This table illustrates that the concrete cured in seawater always has a lower Young's modulus than the reference concrete cured under room conditions.

In Fig. 7 the Young's modulus values have been plotted as a function of the compressive strength of the concrete; also plotted is the theoretical curve given



FIG. 6-Influence of the curing conditions on the compressive strength at different ages.

			W/C	Ratio		
	0.45		45 0.50		0.	55
	Seawater	Reference	Seawater	Reference	Seawater	Reference
Tested at 14 days	26.0	32.5	23.6	31.1	27.8	31.6
28 days	27.3	35.7	25.4	35.8	28.3	34.6

TABLE 7—Young's modulus measurements, GPa, under different W/C ratios and curing conditions.



by American Concrete Institute (ACI) Standard 318-77 [1]. In this figure it can be seen that the relationship between the compressive strength and the Young's modulus is quite different depending on the curing conditions of the concrete. Concrete cured in seawater at  $0^{\circ}$ C ( $32^{\circ}$ F) has a lower Young's modulus than concrete of the same compressive strength cured under standard conditions. For example, for 35 MPa compressive strength (5075 psi) the difference is about 6 GPa ( $10^{6}$  psi), approximately 25%.

#### **Second Simulation**

Because the results of this first simulation had shown that concrete could gain compressive strength when cured in seawater at 0°C (32°F), a second simulation under field conditions was carried out in August 1982 in Nanisivik, in the extreme north of Baffin Island (Fig. 8).

Concrete specimens were cast and cured at ambient temperature for 6, 9, and 12 h, and then were immersed in seawater according to the testing program shown in Table 8. These specimens were tested at 1, 4, and 56 days,



FIG. 8—Nanisivik location.

	Initial Curing	Num Va	Total			
Specimen Size	Temperature	1	7	28	56	Specimens
100 by 200 mm	6 h	3	3	3	3	12
(4 by 8 in.)	9 h	3	3	4	4	14
•	12 h	3	3	3	3	12
	reference					
	specimens	3	3	3	3	12
150 by 300 mm	6 h	3	3			6
(6 by 12 in.)	9 h	3	3			6
•	12 h	3	3			6
	reference					
	specimens	3	3	3	3	12

TABLE 8—Testing program of the Nanisivik experiment.

along with a series of specimens cured at room temperature. The seawater composition at Nanisivik is given in Table 9; it is almost the same as the seawater composition given in the handbook in Ref 2.

The concrete aggregates, a crushed stone (0 to 20 mm) and a natural sand, had a combined grain size distribution within the limits suggested by Canadian Standards Association (CSA) Standard A23-1 (Fig. 9) [3]. The amount of aggregates used in the mix was measured by volume because no scales were available; a Type 1 cement was added in 40-kg (88-lb) sacks, and the amount of water was adjusted to provide a slump of 110 to 140 mm. The exact volume of water introduced into the mix was measured using graduated barrels and pails in order to know exactly the value of the W/C ratio of the concrete. No additives were used. The approximate composition of the concrete is given in Table 10. Its design strength was 30 MPa (4350 psi).

The concrete specimens were cast in 100 by 200-mm (4 by 8-in.) and 150 by 300-mm (6 by 12-in.) cardboard molds, on Saturday 28 Aug. 1982, at 4:30 P.M. The ambient temperature at that time was about  $5^{\circ}C$  (41°F), and the temperature of the seawater was about  $3^{\circ}C$  (37°F).

The first series of specimens was initially cured for 6 h at ambient tempera-

Chemical Elements	Nanisivik, g/L	Handbook Values [2] for Seawater, g/L
NaC1	25.8	25
MgSo <sub>4</sub>	3.5	4
MgCl <sub>2</sub>	1.2	3
CaCl <sub>2</sub>	0.9	1
KCI	0.6	0.7
Salinity	32	34

TABLE 9—Nanisivik sea water composition.





Component	Batching Composition per 8 m <sup>3</sup>	Approximate Composition per 1 m <sup>3</sup>
Water, L	1180 (exact)	147.50 (248 lb)
Cement Type 10, kg	2240 (exact)	280 (470 lb)
Red stone	3 loader charges	51% by weight
Yellow sand	1 loader charge	49% by weight
Water/cement ratio	0.5	3 (exact)
Air content, %	1.9%	% (exact)
Ambient temperature, °C		5

TABLE 10—Approximate concrete composition for Nanisivik program.

ture and then placed in a special steel cage and submerged in the seawater at 10:30 P.M. The concrete was still plastic at that time. The second and third series of specimens, with curing periods of 9 h and 12 h at ambient temperature, were placed in the seawater the next morning at 1:30 and 4:30 A.M. respectively.

#### Temperature of the Concrete Specimens

The temperature of one specimen of each series was measured during the first 24 h. These temperatures are plotted in Figs. 10, 11, 12, and 13. As can be seen in Table 11, the ambient temperature varied between  $8^{\circ}C$  ( $46^{\circ}F$ ) and  $-2^{\circ}C$  ( $28^{\circ}F$ ) and the seawater temperature between  $-2^{\circ}C$  ( $28^{\circ}F$ ) and  $4^{\circ}C$ 



FIG. 10—Comparison of the temperature of the indoor cured concrete and the 9-h submerged series during the first 24 h.



FIG. 11-Variation of the temperature of the 6-h series specimens.



FIG. 12-Variation of the temperature of the 9-h series specimens.

(39°F), whereas the temperature of the concrete cured at room temperature, about  $15^{\circ}C$  (59°F), rose to  $25^{\circ}C$  (77°F) after 24 h of curing.

Figure 10 shows that the induction period of the cement was about 8 h for the specimens stored at room temperature; by that time, a strong increase in the temperature of the concrete was noticed. A small peak in the temperature in the 9 and 12-h concrete was also observed before their submergence in the seawater at 0°C ( $32^{\circ}$ F). Moreover Figs. 11, 12, and 13 illustrate that because



FIG. 13-Variation of the temperature of the 12-h series specimens.

Time after Casting	Ambient Temperature, °C	Seawater Temperature, °C
2 h, 30 min	4	3
2 h, 45 min	5	3
3 h, 10 min	5	3
4 h, 00 min	8	3
4 h, 45 min	3	
5 h. 30 min	3	1
6 h, 00 min	1	1
6 h, 45 min	1	1
9 h. 00 min	3	2
12 h, 15 min	-2	-2
23 h, 00 min	8	4

TABLE 11—Ambient temperature and seawater temperature during the first 24 h.

of the small size of the specimens, the concrete very rapidly reached the temperature of the seawater.

#### **Compressive Strength Results**

The compressive strength results are recorded in Table 12. It shows that the 24-h compressive strength of the concrete cured in seawater was very low. However, after four days, all of the concrete specimens cured in seawater had reached a compressive strength of about 15 MPa (2175 psi), regardless of their initial curing period at the ambient temperature. This is more than half the strength of the concrete cured at room temperature.

Unfortunately, specimens could not be tested at 28 days because the steel

Initial Curing Period	Cured in Seawater				
	1 Day	4 Days	28 Days	56 Days	After One Winter (365 Days)
6 h	0.9	15.0		36.9	38.7
9 h	1.3	14.1		37.4	39.3
12 h	1.2	14.8		33.6	41.1
Reference	12.0	25.6	41.4	43.2	50.5

 TABLE 12—Compressive strength results, MPa<sup>a</sup> (average of three specimens) of concrete specimens cured in seawater.

<sup>a</sup>Multiply numbers by 145 to obtain pounds per square inch.

cage in which the specimens had been placed was lost because of a corrosion problem; however, two divers were able to recover them before the 56-day testing date. At that time the sea was covered with 200 mm (8 in.) of ice, and the seawater temperature was -1.75°C (29°F) (the equilibrium temperature of freezing seawater). At 56 days the concrete specimens cured in seawater had an average compressive strength of 36 MPa (5220 psi), whereas the room-cured concrete specimens had a 28-day compressive strength of 41.4 MPa (6000 psi).

After one winter of exposure in the Arctic Sea (365 days), all of the concrete specimens had a compressive strength of approximately 40.0 MPa (5800 psi). The reference specimens had an average compressive strength of 50.5 MPa (7320 psi). It can be noticed that in this second simulation the compressive strength of the seawater-cured specimens is consistently lower than that of the room-cured specimens. For the seawater-cured specimens, the difference in compressive strength at 56 days and one year was approximately 5 MPa (750 psi). The design strength of the concrete, 30 MPa (4350 psi), however, had been reached at 56 days for all the specimens.

#### Conclusion

Two sets of experiments simulating the curing conditions of concrete caisson construction in the Arctic have shown that concrete can reach its design strength when cured in Arctic Sea water if it has a minimum of 9 h of initial curing at about  $4^{\circ}C$  (39°F).

During the first few days of such a curing, the compressive strength increases very slowly. However, the 28 and 56-day compressive strengths of specimens cured under such harsh conditions can be about the same as that of companion specimens cured under water at room temperature. The Young's modulus of a concrete cured at such a low temperature, however, is lower than that of a similar concrete cured at room temperature.

The initial temperature of the concrete, its water/cement ratio, and the type of cement are critical factors that significantly influence the initial compressive strength of the concrete.

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## Static and Cyclic Behavior of Structural Lightweight Concrete at Cryogenic Temperatures

**REFERENCE:** Berner, D., Gerwick, B. C., Jr., and Polivka, M., "Static and Cyclic Behavior of Structural Lightweight Concrete at Cryogenic Temperatures," *Temperature Effects on Concrete, ASTM STP 858*, T. R. Naik, Ed., American Society for Testing and Materials, Philadelphia, 1985, pp. 21-37.

**ABSTRACT:** The mechanical behavior of a high-strength, lightweight concrete made with expanded-shale aggregate was determined in the temperature range from  $23^{\circ}$ C ( $73^{\circ}$ F) to  $-196^{\circ}$ C ( $-320^{\circ}$ F). High-strength, lightweight concrete is of particular interest for use in offshore cryogenic containment structures in which the concrete may be subjected to low temperatures and high-intensity cyclic loading simulating 20-year stormwave action. Values of compressive strength, tensile strength, and elastic modulus were determined, with moisture content and cyclic loading serving as key parameters. An evaluation was also made of the behavior of embedded strain gages at cryogenic temperatures. The results indicate that the lightweight concrete performed favorably under the test conditions, with the mechanical properties generally increasing at low temperatures with greater gains for higher moisture contents. Cyclic loading induced relatively minor fatigue damage in the concrete and should not affect the structural performance of an offshore containment structure.

**KEY WORDS:** lightweight concrete, cyclic fatigue, low temperature, mechanical properties, cryogenic temperatures, embedded strain gages, marine concrete, high-intensity cyclic loading, concrete

Concrete is one of the few structural materials commonly used at room temperature that also exhibits excellent behavior at very low temperatures. The use of structural lightweight concrete is particularly beneficial in the offshore containment of cryogenic liquids such as liquefied natural gas (LNG), primarily methane, and liquefied petroleum gas (LPG), primarily propane and butane. These liquefied gases have boiling points of approximately  $-160^{\circ}$ C

<sup>1</sup>Associate instructor and professors of civil engineering, respectively, University of California, Berkeley, CA 94720; Berner is presently an engineer at Ben C. Gerwick, Inc., San Francisco, CA.  $(-260^{\circ}\text{F})$  for methane and about  $-42^{\circ}\text{C}$   $(-44^{\circ}\text{F})$  and  $-1^{\circ}\text{C}$   $(30^{\circ}\text{F})$  for propane and butane, respectively.<sup>2</sup> By locating cryogenic containment structures offshore, these volatile liquids would be isolated from the public, and the regasified liquid could be safely transferred ashore by means of a pipeline. From a marine standpoint, lightweight concrete is desirable because of its excellent durability and low maintenance, whereas from a cryogenic standpoint it is desirable for its insulating as well as its elastic and thermal strain characteristics.

The research reported here was undertaken in conjunction with a continuing research program to investigate the behavior of high-strength, prestressed, lightweight concrete slab elements subjected to low temperatures and high-intensity cyclic membrane stress such as would be encountered during a 20-year storm in the North Atlantic. Preliminary results of the composite behavior of prestressed lightweight concrete were previously presented [1]; this report focuses on the mechanical properties of plain lightweight concrete subjected to low temperatures and cyclic fatigue, as well as the methods used to determine these properties.

Previous investigators [2-5] have found that the mechanical properties of moist concrete increase from one to two times at cryogenic temperatures. All of these previous studies were conducted under static conditions and did not investigate the behavior of high-strength lightweight concrete under conditions that might be encountered offshore. The results obtained from this study reaffirms the excellent cryogenic behavior of concrete reported by previous investigators and extend their findings to demonstrate that the mechanical behavior of high-strength lightweight concrete compares favorably with that of standard-weight concrete at low temperatures. Furthermore, this study indicates that the influence of cyclic loading at cryogenic temperatures does not significantly affect the structural performance of a concrete having a moisture content expected in an offshore concrete containment vessel.

Moisture content plays a key role in the behavior of concrete at very low temperatures. Formation of ice within the pores and capillaries of the hardened cement paste contributes to the increases in strength and elastic modulus observed at very low temperatures. Thus, saturated concrete exhibits larger strength gains than dry concretes at low temperatures. Moisture content also strongly influences the cryogenic freeze-thaw durability of concrete, with moisture content above the critical degree of saturation resulting in much greater deterioration than drier concretes. An offshore concrete cryogenic containment vessel would be constructed of high-quality/low-permeability concrete, and most likely with the inside face of the hull exposed to dry, circulating inert gas for the detection of flammable gas leaks through the insulation surrounding the primary containment tank. This combination of low-permeability concrete and active drying ensures that the critical inside

<sup>&</sup>lt;sup>2</sup>Original measurements were taken in English units.

surface of the concrete hull would be below the critical point even for uncoated concrete beneath the surface of the water.

#### **Test Procedures**

The testing program included an accelerated history of cyclic compressive loading at temperatures ranging from  $21^{\circ}C(70^{\circ}F)$  to  $-190^{\circ}C(-310^{\circ}F)$  performed on plain structural lightweight concrete cylinders. The histogram of cyclic loading, shown in Fig. 1, is intended to approximate the 20-year stormwave loading on a floating cryogenic containment vessel. The histogram indicates a nominal precompression of 37.5% of the 28-day compressive strength, which was induced by the testing machine so that the cylinders would more closely approximate the concrete in a prestressed concrete hull. The specimens were cycled from higher to lower compressive stress to simulate axial tension and compression in a prestressed concrete hull due to alternating hogging and sagging waves. These tests were performed in a 1.33-MN (300-kip) capacity MTS testing machine.

All the specimens were brought into thermal equilibrium at a given temperature before application of the histogram of cyclic loading shown in Fig. 1. The cooling rate used was  $0.55^{\circ}$ C/min (1°F/min) to minimize the effect of thermal shock. Cyclic loading was applied at 10 Hz for the first 11 000 low-



FIG. 1-Histogram of cyclic loading.

level cycles, at 1 Hz for the next 110 cycles, and at approximately 0.5 Hz for the remaining high-intensity cycles.

Moisture content was also a key parameter in this investigation. Rostasy, Schneider, and Wiedemann [6] have shown that concrete stored at a relative humidity greater than 85% will exhibit substantially greater strength loss with cryogenic thermal cycling than concrete exposed to a lower relative humidity. Research in the current investigation focused on air-dry and moist concretes with only a few specimens tested in the oven-dry state.

Tests in compression were performed using the low-temperature setup shown in Fig. 2. The insulated cooling chamber was mounted directly in the testing machine, and load was applied to the specimens through closely fitting openings in the top and bottom of the chamber. Liquid nitrogen, which has a boiling point of about  $-196^{\circ}C$  ( $-320^{\circ}F$ ), was used to cool the test chamber. The liquid nitrogen was carried into the chamber by means of a copper cooling coil and was sprayed into the chamber as a fine mist which readily vaporized. Temperature was monitored by thermocouples embedded within a control specimen positioned behind the test specimen in the cooling chamber.



FIG. 2-Low-temperature test setup.

Externally mounted linear variable differential transformers (LVDTs) were used to monitor the movement of the loading platens. In order to calibrate the external LVDTs, an aluminum cylinder of known cryogenic behavior was subjected to the same loading conditions at low temperatures as were the concrete specimens.

Splitting tension tests were performed in the same cooling chamber used for the compression tests (Fig. 2). A control specimen with embedded thermocouples to monitor temperatures was also located in the cooling chamber. No cyclic tests were performed on the tensile strength specimens.

Supplementary tests were also performed to determine the effectiveness of different resistance-type embedded strain gages at cryogenic temperatures. Five types of resistance strain gages were embedded within three 152 by 406-mm (6 by 16-in.) concrete cylinders. The five types of gages were (1) a 203-mm (8-in.) modified A-8 Carlson strain meter containing no oil except for a thin coating on all metal parts to prevent corrosion; (2) a 102-mm (4-in.) M-4 Carlson strain meter modified to replace the normal petroleum-based oil with a low-viscosity oil; (3) a 102-mm (4-in.) BLH-Bakelite encased strain gage; (4) a 152-mm (6-in.) Ailtech embedded strain gage; and (5) two 102-mm (4-in.) Micro-Measurement foil strain gages, each prebonded with epoxy between two concrete prisms to form a sandwich before being cast in the specimens. To monitor temperature, thermocouples were embedded along the length and diameter of the cylinders.

#### **Concrete Mix Design and Test Specimens**

A single, high-strength lightweight concrete mix was used in all phases of this test program. The mix design is summarized in Table 1. An expanded shale was used for the coarse aggregate with a maximum size of aggregate (MSA) of 9.5 mm ( $\frac{3}{6}$  in.), while the fine aggregate was of a natural sand with a fineness modulus of 2.49. The mix had a nominal 44.8-MPa (6500-psi)

Cement, kg/m <sup>3</sup> (lb/yd <sup>3</sup> )	433	(730)
Fly ash, $kg/m^3$ (lb/yd <sup>3</sup> )	94	(159)
Water, $kg/m^3$ ( $lb/yd^3$ )	186	(313)
Fine aggregate, kg/m <sup>3</sup> (lb/yd <sup>3</sup> )	752	(1268)
Coarse aggregate, kg/m <sup>3</sup> (lb/yd <sup>3</sup> )	463	(781)
Admixtures		
Water reducing, $mL/m^3$ (oz/yd <sup>3</sup> )	1271	(33)
Air entraining, $mL/m^3$ (oz/yd <sup>3</sup> )	282	(7.5)
Water/(cement + fly ash)	0.35	
Slump, cm (in.)	7.6	(3)
Air content, %	4.5	
Unit weight, kg/m <sup>3</sup> (lb/yd <sup>3</sup> )	1922	(120)
Nominal compressive strength, MPa (psi)	44.8	(6500)

TABLE 1—Lightweight concrete mix.

28-day compressive strength, with a water-to-cementitious-material ratio of 0.35.

Two sizes of specimens were used for the cyclic loading and splitting tensile strength tests, however, the test results were normalized to indicate the results for 152 by 305-mm (6 by 12-in.) concrete cylinders. Compressive strength and elastic modulus in both cycled and uncycled conditions were obtained using 102 by 203-mm (4 by 8-in.) cylinders. To ensure uniform loading at low temperatures, the ends of the specimens were ground off and made plane within 0.050 mm (0.002 in.) and perpendicular within  $0.5^{\circ}$  of their axis. Splitting tensile strength was determined only for the uncycled state using 76 by 152-mm (3 by 6-in.) cylinders.

These specimens were tested at ages from 90 to 150 days. They were cured to three moisture states: (1) moist specimens were continuously fog-cured until testing; (2) air-dry specimens were fog-cured for 7 days and then maintained at 50% relative humidity until testing; (3) oven-dry specimens were fog-cured for 80 days and then oven dried at  $110^{\circ}C$  ( $230^{\circ}F$ ) for 25 days and sealed to prevent moisture gain. The three 152 by 406-mm (6 by 16-in.) cylinders cast for evaluation of embedded strain gages were sealed with Saran wrap (polyvinylidene chloride) after demolding, and were considered to be moist at the time of the test.

#### **Test Results**

The high-strength, lightweight concrete studied in this program showed similar but somewhat lower increases in strength and modulus of elasticity than results reported elsewhere [7-9] for standard-weight concrete at cryogenic temperatures. The effects of cyclic loading at low temperatures were less pronounced for air-dry concrete than for moist concrete; however, in no case did the cyclic loading significantly affect the mechanical properties of the concrete. The influence of cyclic loading on air-dry concrete was generally more pronounced at room temperature than at low temperatures. Poisson's ratio was also measured, and it changed uniformly between 0.21 and 0.25 from room temperature down to  $-165^{\circ}$ C ( $-265^{\circ}$ F). The compressive strength, elastic modulus, and tensile strength of oven-dry concrete showed little or no change in going from room temperature to low temperatures; thus, these results are not shown in Figs. 3, 4, and 5. The shaded areas in these figures indicate data scatter.

#### **Compressive Strength**

Compressive strength tests were performed at low temperatures on both cycled and uncycled specimens. Figure 3 shows the results for both moist and air-dry specimens. The strength of uncycled moist specimens increased by approximately 80%, while air-dry concrete increased by more than 30% at








cryogenic temperatures. In all cases, cyclic loading slightly decreased the compressive strength. However, air-dry concrete was not as influenced by cyclic loading at low temperatures as moist concrete. Oven-dry concrete followed the same trend as air-dry concrete. This difference in the response of air-dry and moist concrete to cyclic loading may be attributed to the response of ice to cyclic loading, as discussed in the section on the influence of ice.

#### Elastic Modulus

As shown in Fig. 4, the modulus of elasticity of moist concrete increased by approximately 75%, while air-dry concrete increased by about 65% at cryogenic temperatures. The cyclic loading had a relatively minor effect on the modulus of elasticity of both moist and air-dry lightweight concrete at low temperatures. However, slightly larger losses in stiffness were recorded for both moist and air-dry concrete at room temperature than at cryogenic temperatures.

# Splitting Tensile Strength

Cyclic loading tests were not performed on splitting tension specimens. The splitting tensile strengths showed a marked increase between  $-6.7^{\circ}C$  (20°F) and  $-87^{\circ}C$  ( $-125^{\circ}F$ ), and then gradually decreased with decreasing temperature. At low temperatures the moist specimens increased approximately 80% in tensile strength, while the air-dry specimens increased by about 50%. Oven-dry specimens showed little or no increase in tensile strength at low temperatures.

# Evaluation of Embedded Strain Gages

The results of tests to evaluate the behavior of embedded strain gages are shown in Figs. 6 and 7. Figure 6 shows calibration curves for interpreting the output of the embedded strain gages. These curves represent the indicated strains at zero load at several low temperatures. These indicated strains include the combined effects of differential thermal contraction between the strain gage and the concrete, as well as the thermally induced change in resistance of the sensing element. As illustrated in Fig. 6, the BLH-Bakelite encased gage had a very large correction factor and is not recommended for cryogenic use. The epoxy-bonded foil gages are also not recommended because of difficulties encountered with the epoxy bonding agent. Both the Ailtech and the modified A-8 Carlson embedded strain gages worked well, however, the oil-filled M-4 Carlson gage encountered problems, apparently from stiffening of the low-viscosity oil.

Figure 7 presents a comparison of values of elastic modulus for moist concrete at low temperatures as determined by external LVDTs mounted to the



FIG. 6-Effect of temperature on indicated strain of embedded gages at zero load.

platens of the testing machine and three types of embedded strain gages. The line representing the LVDT values is an average of the data for uncycled moist concrete shown in Fig. 4. The envelope bounding the shaded area represents the range of experimental values obtained by the different strain gages. The results indicate that selected types of embedded resistance gages work well and can be used to monitor strains in concrete at low temperatures.

Figure 8 presents the effect on the elastic stiffness of one thermal cycle from room temperature down to  $-179^{\circ}C$  ( $-290^{\circ}F$ ) and back to room temperature. This test was performed on moist lightweight concrete using an embedded Ailtech gage. The degradation shown after one cycle agrees with findings of other researchers [6,8] and is in the order of magnitude of degradation TEMPERATURE EFFECTS ON CONCRETE

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FIG. 8-Effect of one thermal cycle on stiffness of concrete.

observed in elastic modulus from cyclic loading at cryogenic temperatures. It is expected that this degradation caused by thermal cycling will diminish with each subsequent cycle.

## **Influence** of Freeze-Thaw

Previous investigators [6,8] have studied the influence of freeze-thaw cycles down to cryogenic temperatures and have shown significant differences from the influence of normal freeze-thaw cycles around 0°C ( $32^{\circ}$ F). Tognon [8] demonstrated that concrete undergoing thermal cycling at 85% relative humidity showed little degradation in stiffness and flexural strength and also showed little or no change in compressive strength. As mentioned earlier, Rostasy, Schneider, and Wiedemann [6] confirmed these findings and extended them to demonstrate that concretes with moisture contents about 85%showed much greater strength losses than drier concretes. The rate of cooling also has a pronounced influence, with higher cooling rates introducing much more degradation. Freeze-thaw cycles down to cryogenic temperatures can induce several times the degradation as standard freeze-thaw cycles at about  $0^{\circ}C$  (32°F).

# **Influence of Ice**

Because of the formation of ice within the capillaries and gel-pores, moisture content has a critical influence on the mechanical properties of concrete at cryogenic temperatures. Adsorbed water has a lower freezing point than bulk water. The smaller the size of the pore, the lower the freezing point of the adsorbed water because of the increase in surface energy per unit volume. All of the adsorbed water will be frozen upon reaching a temperature of from  $-80^{\circ}$ C to  $-95^{\circ}$ C ( $-110^{\circ}$ F to  $-140^{\circ}$ F) [10,11]. The exact relationship between the mechanical properties of concrete and frozen adsorbed water is complex, which may account for the variations in mechanical properties reported in this study and by other researchers [7,9,12].

The primary effect of the ice may be the reduction of stress concentrations within the concrete by filling the discontinuities formed by pores and capillaries. Other important factors include the following:

1. Ice itself increases in strength with decreasing temperature. Haynes [13] reports an increase of compressive strength of ice from 6.9 MPa (1000 psi) at  $0^{\circ}$ C (32°F) to approximately 48.3 MPa (7000 psi) at  $-55^{\circ}$ C ( $-67^{\circ}$ F).

2. The coefficient of thermal contraction/expansion of ice varies greatly with temperature, ranging from approximately  $9 \times 10^{-6/\circ}$ C at  $-196^{\circ}$ C (16.2  $\times 10^{-6/\circ}$ F at  $-321^{\circ}$ F) to 55  $\times 10^{-6/\circ}$ C at  $0^{\circ}$ C (99  $\times 10^{-6/\circ}$ F at  $32^{\circ}$ F), as reported by Yen [14].

3. As noted by Radjy and Richards [15], the internal friction of hardened cement paste exhibits a sharp increase or "peak" in the temperature range from  $-60^{\circ}$ C ( $-76^{\circ}$ F) to  $-160^{\circ}$ C ( $-256^{\circ}$ F), which corresponds to a sharp increase in the elastic modulus of a saturated paste. This internal friction "peak" occurs also for ice at about the same temperature as determined by Vassoille, Mai, and Perez [16].

4. Helmreich [17] has reported a decrease in the elasticity of ice between  $-120^{\circ}C$  ( $-184^{\circ}F$ ) and  $-190^{\circ}C$  ( $-310^{\circ}F$ ), with a minimum elasticity at about  $-170^{\circ}C$  ( $-270^{\circ}F$ ). As shown in Fig. 9, this decrease or "dip" does not occur for cooling rates faster than  $0.7^{\circ}C/min$  ( $1.25^{\circ}F/min$ ) or for heating rates faster than  $0.3^{\circ}C/min$  ( $0.5^{\circ}F/min$ ). A similar dip in compressive strength was observed for saturated concrete in the same temperature range by Monfore and Lentz [7]; Yamane, Kasami, and Okuno [9]; and in this



FIG. 9—Compressive strength of lightweight concrete and elasticity of ice at low temperatures.

study. All of these researchers used a slow cooling rate of less than  $0.7^{\circ}$ C/min (1.25°F/min). Rostasy and Wiedemann [12], who used a relatively high rate of cooling of 2°C/min (3.6°F/min), showed no decrease in compressive strength for saturated specimens. The data given in Fig. 9 clearly demonstrate the influence of ice on the strength of concrete and the effects of rate of cooling.

Air-dry specimens do not exhibit such a sudden decrease or dip in compressive strength since moisture is present in only the very narrow capillaries and gel pores having high surface energy. Eldrup and Mogensen [18] have related this dip in elasticity to the migration of vacancies and dislocations of the ice crystal lattice. This may account for the smaller influence of cyclic loading at low temperatures on air-dry concrete than on moist concrete. Cyclic loading may provide the activation energy required for the migration of dislocations. These dislocations may be less numerous or not as free to migrate within smaller capillaries and gel pores, where the adsorbed water resides within airdry concrete. Thus, after freezing, the elasticity of this ice within the smaller capillaries and gel pores may be less susceptible to "softening" than that of the bulk ice found in the larger capillaries and voids of frozen moist concrete.

# Conclusions

1. The high-strength, lightweight concrete used in this study exhibited increases in mechanical properties that were similar to but lower than those reported by other investigators for standard-weight concrete.

2. The moisture content of concrete has a significant influence on strength and elasticity, with moist concrete exhibiting much greater increases at low temperatures than air-dry concrete.

3. Air-dry concrete exhibits greater resistance to cyclic fatigue damage at low temperatures than at room temperature, presumably because of the increase in strength at low temperatures.

4. Air-dry concrete is not influenced as much as saturated concrete by cyclic loading at cryogenic temperatures, presumably because of the effect of cyclic loading on ice.

5. Selected resistance-type embedded strain gages can be used effectively to measure elastic strain at cryogenic temperatures.

6. High-strength, lightweight concrete performs well under conditions that may be encountered in the offshore containment of cryogenic liquids.

# **Recommendations for Future Research**

Further research and considerations requiring additional investigation include the following:

(a) the influence of cooling rate of concrete on its compressive and tensile strength as well as on its elastic modulus;

(b) the influence of cryogenic freeze and thaw cycles on high-strength concrete at different moisture conditions;

(c) the influence of cyclic loading on the mechanical properties of ice at cryogenic temperatures; and

(d) the influence of cyclic loading of concrete up to  $10^8$  cycles.

# **Acknowledgments**

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# Performance of Dolostone and Limestone Concretes at Sustained High Temperatures

**REFERENCE:** Carette, G. G. and Malhotra, V. M., "**Performance of Dolostone and Limestone Concretes at Sustained High Temperatures**," *Temperature Effects on Concrete, ASTM STP 858*, T. R. Naik, Ed., American Society for Testing and Materials, Philadelphia, 1985, pp. 38-67.

**ABSTRACT:** This report gives results of a laboratory study undertaken to evaluate the relative performance of limestone and dolostone aggregate concretes under sustained exposure to high temperatures. For each type of concrete, three water/cement (W/C) ratios ranging from 0.33 to 0.60 were investigated. The test specimens were exposed for up to four months to temperatures ranging from 76 to 600°C. The conditioning of the specimens prior to any temperature exposure consisted of moist curing for 28 days followed by storage under room conditions for 26 weeks.

The test results show the dolostone aggregate to be unstable under a sustained temperature of  $150^{\circ}$ C, confirming the results of a previous investigation. Such instability is again attributed to the slow oxidation of the pyrite contained in some of the aggregate particles. The resulting expansion causes disintegration of the aggregate and rupture of the concrete. Under similar exposure, concrete made with a limestone aggregate is found to be unaffected.

Except for these results, the loss of compressive strength of the specimens under exposure is generally independent of the type of aggregate and is proportional to the temperature. At temperatures of 150°C and higher, an increase in the length of exposure from 48 h to four months results in further decrease in strength. In all cases, however, any major strength loss is found to occur within the first month of exposure. In general, the leaner concretes are slightly less affected than the richer concretes in terms of relative strength loss after exposure.

Both pulse velocity and resonant frequency measurements appear to provide excellent means of monitoring the compressive strength loss during exposure.

**KEY WORDS:** high temperatures, concrete, compressive strength, pyrites, dolostone aggregate, limestone aggregate, pulse velocity, resonant frequency

In 1979 CANMET initiated a research project to develop long-term strength data on concrete exposed to sustained high temperatures. Results of the first phase of this study, dealing with the performance of concrete made

<sup>1</sup>Materials engineer and head, Construction Materials Section, respectively, Mineral Sciences Laboratories, CANMET, Energy, Mines, and Resources Canada, Ottawa, Canada K1A 0G1. with normal portland cement, normal portland cement and slag, or normal portland cement and fly ash at sustained high temperatures, have been reported elsewhere [1]. Briefly, the following conclusions were reached:

1. In general, the incorporation of fly ash and slag in the concrete had little effect on the mechanical properties of concrete after exposure to sustained high temperatures. This was true regardless of the exposure temperature and the water-to-cementitious-materials ratio.

2. Some dolomitic aggregate particles were found to be unstable at a sustained temperature of 150°C, leading to the disruption of the concrete regardless of the water-to-cement (W/C) ratio or water-to-cementitious-materials ratio. This unusual behavior of aggregate at 150°C appears to be due to the presence of iron sulfide.

3. The significant changes in the mechanical properties of the concrete under long-term exposure occurred within the first month, except for the 150°C temperature exposure when the sustained temperature effects were masked by the unusual instability of the coarse aggregate.

This phase of the investigation was undertaken to compare the performance of concrete made with dolostone aggregate as used in the previous phase with that of concrete made with a reference limestone at exposure temperatures ranging from 75 to 600°C.

#### Scope

Two types of concrete were investigated. One type consisted of normal portland cement, limestone as coarse aggregate, and natural sand as fine aggregate. The second type consisted of normal portland cement, dolostone as coarse aggregate, and natural sand as fine aggregate. The W/C ratio, by weight, ranged from 0.33 to 0.60. The concretes were exposed to temperatures of 75, 150, 300, 450, and 600°C, and the duration of exposure was 48 h, one month, and 4 months.

The conditioning of the specimens prior to any temperature exposure involved moist curing for 28 days followed by storage at normal room temperature for 26 weeks. The compressive strengths were determined after 28 days of moist curing, and then before and after different periods of temperature exposure. Changes in weight, pulse velocity, and resonant frequency of the specimens were also monitored at the same time.

#### **Concrete Mixes**

A series of six concrete mixes, each  $0.1 \text{ m}^3$  in volume, was prepared for the investigation.

# Materials

Cement—A commercially available normal portland cement, ASTM Type I (CSA Type 10), was used. A summary of its physical and chemical properties is given in Table 1.

Aggregates—Two types of -19-mm crushed coarse aggregates were investigated. One type consisted of high-calcite limestone and the other was mostly dolostone. The mineralogical composition (basically a limy to arenaceous dolostone with shaly layers) of the latter has been described in detail elsewhere [2]. Both of these aggregates were from sources approved by the local highway department for use in concrete.

The fine aggregate was a local natural sand. Each aggregate was separated into fractions of specific size and recombined to a predetermined specifica-

Description of Test	Portland Cement <sup>a</sup> (ASTM Type 1)
Physical tests—general	
Time of set (Vicat needle)	
Initial	2h, 04 min
Final	3h, 52 min
Fineness	
75 μm (passing), %	97.0
45 $\mu$ m (passing), %	85.9
Surface area, Blaine, m <sup>2</sup> /kg	352
Soundness, autoclave expansion, %	0.001
Physical tests-mortar strength	
Compressive strength, MPa, of 51-mm cubes at	
3 days	22.7
7 days	30.6
28 days	39.8
Chemical analysis, %	
Insoluble residue	0.14
Silicon dioxide $(SiO_2)$	21.54
Aluminum oxide $(Al_2O_3)$	4.84
Ferric oxide ( $Fe_2O_3$ )	2.10
Calcium oxide (CaO), total	64.10
Magnesium oxide (MgO)	2.30
Sulfur trioxide $(SO_3)$	3.97
Loss on Ignition	0.73
Compound composition, %	
C <sub>3</sub> S	50.4
C <sub>2</sub> S	23.7
C <sub>3</sub> A	9.3
C₄AF	6.4

TABLE 1—Physical properties and chemical analysis of cement.

<sup>a</sup> Manufacturer's data.

tion to provide a uniform grading for each mix. The grading and physical properties of the aggregates are shown in Tables 2 and 3.

## Mix Proportioning

The detailed mix proportions are given in Table 4. For each type of aggregate, three mixes were made with W/C ratios of 0.33, 0.45, and 0.60. All the mixes were proportioned for a nominal slump of 60 mm. No air-entraining agent or other admixtures were used. The room-dry coarse and fine aggregates were soaked in water for 24 h before they were used, and the amount of mixing water was adjusted according to the water absorbed.

#### Mixing Procedure

All the concrete mixes were prepared in a laboratory countercurrent mixer with the total mixing time kept constant at 6 min.

## **Properties of Fresh Concrete**

The properties of freshly mixed concrete, that is, slump, unit weight, and air content, are shown in Table 4.

Coarse A	ggregate	Fine Aggr	egate
Sieve Size, mm	Cumulative Percentage Retained	Sieve Size	Cumulative Percentage Retained
	0	No. 4 (4.75 mm)	0
12.5	33.3	No. 8 (2.38 mm)	10.0
9.5	66.3	No. 16 (1.19 mm)	32.5
4.75	100.0	No. 30 (600 μm)	57.5
		No. 50 (300 µm)	80.0
		No. 100 (150 µm)	94.0
		pan	100.0

TABLE 2-Grading of aggregates.

TABLE 3—Physical properties of coarse and fine aggregates.

Physical Property	Dolostone	Limestone	Natural Sand
Bulk specific gravity	2.71	2.70	2.68
Absorption, %	1.10	0.68	1.20

			Mi	x Propor	tions	Propertie	es of Fresh	Concrete
Type of Cement	Mix No.	Cement Content, kg/m <sup>3</sup>	Water Content, kg/m <sup>3</sup>	W/C <sup>a</sup>	A/C <sup>a</sup>	Slump, mm	Unit Weight, kg/m <sup>3</sup>	Entrapped Air, %
Limestone	1	207	125	0.60	7.26	65	2400	2.1
	2	285	128	0.45	5.05	65	2420	2.0
	3	396	130	0.33	3.37	40	2435	2.0
Dolostone	4	209	126	0.60	7.28	50	2430	2.0
	5	287	129	0.45	5.07	70	2450	1.9
	6	403	133	0.33	3.39	40	2485	1.9

TABLE 4—Mix proportions and properties of fresh concrete.

<sup>a</sup> All the ratios are by weight.

#### **Preparation and Curing of Test Specimens**

Forty-eight 102 by 203-mm cylinders were prepared for each mix. All the specimens were cast in steel molds and compacted on a vibrating table. The specimens were kept in their molds under wet burlap for 24 h, after which they were demolded and stored under moist-curing conditions for 28 days. During this period, the ends of the specimens were lapped to obtain a smooth surface to facilitate pulse velocity measurements. After 28 days, all the specimens were stored under laboratory air-drying conditions at a relative humidity of about 50% for a period of 26 weeks. Following this, the specimens were exposed to elevated temperatures.

#### **Conditions of Exposure**

The exposure temperatures were 75, 150, 300, 450, and  $600^{\circ}$ C. The periods of exposure were 48 h, one month, and four months. For all the exposures, the heating or cooling rate did not exceed 20 degrees Celsius per hour. The tests were carried out under unrestrained moisture conditions, that is, the moisture was free to move out of the concrete as well as to escape from the heating chambers.

# **Heating Equipment**

For all the exposures, electrically heated kilns designed for temperatures up to 1200°C were used. Each kiln was heated by means of exposed heating elements laid on the refractory walls of the inside chamber and arranged to permit separately controlled heating zones. The chambers, approximately 0.9 by 1.2 by 1.8 m high, were divided into four levels to accommodate the test specimens. Before any exposure tests, the heat distribution in the chambers was determined under the proposed temperatures of exposure and for different conditions of loading. Excessive temperature gradients were generally observed within a specific area near the heating coils. However, for any temperature or loading condition, the greater part of the chambers, including the loading areas for the tests, was found to be under a maximum temperature gradient of about 3%.

For the exposure tests, four Chromel/Alumel thermocouples connected to a multipoint recorder were installed at different locations within the loaded area of each chamber to provide for continuous monitoring of the temperature.

#### **Testing of Specimens**

To evaluate the effects of the elevated temperatures on the concrete, the following tests were performed on the specimens before and after heat exposure in accordance with the testing schedule given in Table 5.

- (a) visual examinations,
- (b) weight determinations,
- (c) pulse velocity measurements,
- (d) resonant frequency measurements, and
- (e) compression tests.

All the tests were carried out at room temperature and, as far as possible, in accordance with the ASTM procedures.

# **Test Results**

The strength data given in Tables 6 and 7 are plotted in Figs. 1 to 6. The results of weight, pulse velocity, and resonant frequency determinations are summarized in Tables 8 to 10.

# **Discussion of Test Results**

Both limestone and dolostone concretes had about the same compressive strength at time of exposure, with values ranging from 43 MPa for concretes with a W/C ratio of 0.60 to 71 MPa for those with a W/C ratio of 0.33. The percentage of free moisture then present in any specimen (as determined on companion samples) ranged from 2.1% for the lean concretes to 3.4% for the rich ones.

# Exposure at 75°C

At 75°C, the removal of free moisture from the specimens was found to proceed gradually, especially during the early stages of exposure. For exam-

					Testing Sc of T	hedule an est Speci	nd Number mens	
				Refe	erence		Heat-Expos	ed
Type of Concrete	Mix No.	W/C Ratio (by weight)	Exposure Temperature, °C	28-Day Moist- Cured	6-Month Air- Dried	48 h	1-Month	4-Month
Limestone	1	0.60	reference 75 150 300 450 600	3	3	3 3 3 3 3	3 3 3 3 3	3 3 3 3
	2	0.45	reference 75 150 300 450 600	3	3	3 3 3 3 3	3 3 3 3 3	3 3 3 3
	3	0.33	reference 75 150 300 450 600	3	3	3 3 3 3 3	3 3 3 3 3	3 3 3 3
Dolostone	4	0.60	reference 75 150 300 450 600	3	3	3 3 3 3 3	3 3 3 3 3	3 3 3 3
	5	0.45	reference 75 150 300 450 600	3	3	3 3 3 3 3	3 3 3 3 3	3 3 3 3
	6	0.33	reference 75 150 300 450 600	3	3	3 3 3 3 3	3 3 3 3 3	3 3 3 3

TABLE 5—Testing schedule and number of test specimens.

ple, about 40% of the free moisture in the rich concrete had been driven out after 48 h, in comparison with about 80 and 95% after one and four months, respectively (Table 8).

The test results indicate, in general, substantial reduction in the strength of the specimens after exposure, though the pattern appears somewhat irregular (Table 7). At 48 h, strength losses were generally on the order of 15 to 20%, except for the dolostone concrete with a W/C ratio of 0.60, which showed

			Compressive Reference Co	e Strength of oncrete, MPa	Exposure	Comp Concre	ressive Strer te after Exp	ngth of osure to
Type of Concrete	Mix No.	W/C Ratio (by weight)	28-Day Moist- Cured	6-Month Air- Dried	Temper- ature, °C	48-h Exposure	1-Month Exposure	4-Month Exposure
	1	0.60	32.9	42.8	reference			
Linestone	•	0.00	02.7		75	34.8	39.6	38.3
					150	37.1	34.5	34.6
					300	29.7	26.8	21.7
					450	22.3	20.3	14.4
					600	13.1	10.8	
	2	0.45	47 9	63.3	reference			
	2	0.45		05.5	75	52.5	55 7	52.9
					150	51.8	49.4	45.9
					300	41.2	36.0	27.7
					450	27.8	25.4	16.0
					600	15.8	12.1	
	3	0.33	577	71.0	reference			
	5	0.55	57.7	/1.0	75	55.6	58.8	60.1
					150	56.2	55.2	50.8
					300	40.3	39.7	31.6
					450	26.5	25.8	16.1
					600	15.2	14.3	
Dolostone	4	0.60	31.4	42.7	reference			
Delestene	•	0.00	0,111		75	42.4	43.3	39.6
					150	39.2	36.8	b
					300	30.6	27.1	25.7
					450	23.8	20.3	15.8
					600	16.3	11.5	
	5	0.45	48.1	64.2	reference			
	Ũ	0110		• ••=	75	55.4	60.9	53.7
					150	54.6	49.4	Ь
					300	43.4	35.7	31.8
					450	32.2	25.1	19.7
					600	20.0	13.3	•••
	6	0.33	57.6	71.2	reference			
		0.00	0.10		75	61.8	67.6	59.8
					150	63.1	57.5	Ь
					300	45.8	37.3	32.9
					450	32.8	24.6	19.3
					600	20.3	12.9	

 TABLE 6—Summary of compressive strength test results of reference concrete and concrete after exposure to high temperatures.

<sup>a</sup> Twenty-eight-day moist cured followed by six months of air drying.

<sup>b</sup> Specimens severely damaged.

little change. After one-month exposure, there was a partial recovery of strength in all cases; this was followed by a reversal, which after four months resulted in total strength losses from about 10 to 15% for both types of concrete.

The magnitude of strength losses observed at this temperature exposure is somewhat surprising. One possible explanation is that, even at this relatively

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Exposure	Tupe		W/C	Compressi of Reference	ve Strength as a ce Strength before	Percentage re Exposure
Temperature, °C	of Concrete	Mix No.	Ratio (by weight)	48-h Exposure	1-Month Exposure	4-Month Exposure
75	limestone	1	0.60	81.3	92.5	89.5
		2	0.45	82.9	88.0	83.6
		3	0.33	78.3	82.8	84.6
	dolostone	4	0.60	99.3	101.4	92.7
		5	0.45	86.3	94.9	83.6
		6	0.33	86.8	94.9	84.0
150	limestone	1	0.60	86.7	80.6	80.8
		2	0.45	81.8	78.0	72.5
		3	0.33	79.2	77.7	71.5
	dolostone	4	0.60	91.8	86.2	Ь
		5	0.45	85.0	76.9	ь
		6	0.33	88.6	80.8	Ь
300	limestone	1	0.60	69.4	62.6	50.7
		2	0.45	65.1	56.9	43.8
		3	0.33	56.8	55.9	44.5
	dolostone	4	0.60	71.5	63.5	60.2
		5	0.45	67.6	55.6	49.5
		6	0.33	64.3	52.4	46.2
450	limestone	1	0.60	52.1	47.4	33.6
		2	0.45	43.9	40.1	25.3
		3	0.33	37.3	36.3	22.7
	dolostone	4	0.60	55.7	47.5	37.0
		5	0.45	50.2	39.1	30.7
		6	0.33	46.1	34.6	27.1
600	limestone	1	0.60	30.6	25.2	•••
		2	0.45	25.0	19.1	
		3	0.33	21.4	20.1	•••
	dolostone	4	0.60	38.2	26.9	
		5	0.45	31.2	20.7	
		6	0.33	28.5	18.1	•••

TABLE 7-Summary of residual<sup>a</sup> compressive strength after exposure to high temperatures.

<sup>a</sup> Expressed as percentages of strength before exposure.

<sup>b</sup> Specimens severely damaged.

moderate temperature, some incompatibility in the dimensional changes of the paste and aggregate phases during heating and cooling and from drying shrinkage might have been sufficient to contribute to an overall weakening of the concrete.

This strength loss is somewhat larger than that observed in the first phase of the study; however, this might be related to some difference in the test procedures, such as the use of a relatively more confined environment for the exposure tests in the first phase. A wide scatter of data in this range of tem-



FIG. 1—Compressive strength of limestone concrete after 48 h of exposure to various temperatures.



FIG. 2—Compressive strength of limestone concrete after one month of exposure to various temperatures.



FIG. 3—Compressive strength of limestone concrete after four months of exposure to various temperatures.



FIG. 4—Compressive strength of dolostone concrete after 48 h of exposure to various temperatures.



FIG. 5—Compressive strength of dolostone concrete after one month of exposure to various temperatures.



FIG. 6—Compressive strength of dolostone concrete after four months of exposure to various temperatures.

r.			C, III			Wei	ight of 102 b	y 203-m	m Cylinders, k	6ª		
Temper-	Type	NG-	Ratio	48	-h Exposi	Jre	1-M	onth Exp	osute	4-M	onth Expo	sure
°C °C	on Concrete	No.	(by weight)	Before	After	% Change	Before	After	% Change	Before	After	% Change
75	limestone		0.60	3.851	3.799	1.4	3.847	3.773	1.9	3.906	3.825	2.1
		2	0.45	3.969	3.917	1.3	3.928	3.825	2.6	3.975	3.862	2.8
		3	0.33	4.969	3.989	1.2	4.017	3.903	2.8	3.059	3.930	3.2
	dolostone	4	0.60	3.957	3.906	1.3	3.894	3.820	1.9	3.895	3.817	2.0
		ŝ	0.45	3.985	3.933	1.3	9.359	3.859	2.5	3.956	3.851	2.7
		6	0.33	4.009	3.959	1.3	3.019	3.907	2.8	4.037	3.909	3.2
150	limestone	1	0.60	3.885	3.775	2.8	3.878	3.782	2.5	3.948	3.847	2.6
		2	0.45	3.996	3.852	3.6	4.003	3.866	3.4	3.978	3.842	3.4
		Э	0.33	4.053	3.891	4.0	4.130	3.969	3.9	3.994	3.836	4.0
	dolostone	4	0.60	3.928	3.815	2.9	3.911	3.811	2.6	:	:	:
		ŝ	0.45	3.938	3.798	3.6	3.984	3.850	3.4	:	:	
		9	0.33	4.065	3.904	4.0	4.014	3.856	3.9			

TABLE 8—Summary of weight changes of test specimens after exposure to various temperatures.

•

3.750 3.1	3.761 4.0	3.826 4.7	3.813 3.2	3.891 4.1	3.940 4.8	3.801 3.9	3.768 4.8	3.765 5.5	3.483 10.1	3.535 11.1	3.578 11.5	•	:	:	•	:	::
3.868	3.918	4.015	3.937	4.056	4.137	3.956	3.957	3.985	3.876	3.977	4.042	:	:	:	•	:	:
3.2	4.3	4.9	3.3	4.2	4.8	3.8	4.9	5.7	4.1	5.4	6.3	5.7	7.2	7.1	13.3	13.8	14.4
3.799	3.758	3.817	3.807	3.825	3.793	3.728	3.767	3.809	3.734	3.740	3.798	3.660	3.631	3.756	3.423	3.399	3.457
3.925	3.926	4.013	3.936	3.993	3.986	3.877	3.960	4.040	3.895	3.955	4.052	3.880	3.912	4.044	3.947	3.945	4.039
3.6	4.5	5.0	3.4	4.3	4.9	4.2	5.3	6.2	4.0	5.1	5.9	4.7	5.9	6.4	9.3	10.8	10.9
3.746	3.774	3.785	3.779	3.830	3.838	3.726	3.755	4.803	3.738	3.788	3.821	3.677	3.764	3.734	3.602	3.567	3.612
3.885	3.952	3.985	3.910	4.001	4.035	3.890	3.967	4.053	3.892	3.993	4.062	3.857	3.999	3.990	3.973	4.008	4.053
0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33
1	7	e	4	ŝ	9	1	7	e	4	S	9	1	7	e,	4	ŝ	6
limestone			dolostone			limestone			dolostone			limestone			dolostone		
300						450						600					

<sup>a</sup> Each value is the average of three test results.

L L						Pulse Ve	elocity of 10	2 by 203-i	mm Cylinders,	m/S <sup>4</sup>		
Temper-	Type		Ratio	48	-h Exposi	lite	1-M(	onth Exp(	osure	4-Mc	onth Expo	sure
°C °C	oı Concrete	No.	(oy weight)	Before	After	% Change	Before	After	% Change	Before	After	% Change
75	limestone	-	0.60	4650	4436	4.6	4704	4216	10.4	4671	4008	14.2
		2	0.45	4873	4704	3.5	4885	4312	11.7	4861	4056	16.6
		÷	0.33	4932	4838	1.9	4944	4361	11.8	4944	3908	21.0
	dolostone	4	0.60	4671	4486	4.0	4682	4251	9.2	4944	4089	12.3
		S	0.45	4920	4792	2.6	4944	4496	9.1	4661	4251	13.4
		9	0.33	4968	4850	2.4	5005	4417	11.8	4993	4000	19.9
150	limestone	1	0.60	4737	3992	15.7	4726	3930	16.8	4704	3770	19.9
		7	0.45	4885	4080	16.5	4873	3938	19.2	4896	3827	21.8
		÷	0.33	4932	3946	20.0	4968	3841	22.7	4956	3681	25.7
	dolostone	4	0.60	4704	4056	13.8	4704	3969	15.6	:	:	:
		S	0.45	4932	4278	13.3	4932	4080	17.3	:	:	:
		9	0.33	4993	4172	16.4	5005	3841	23.3	:	:	:

TABLE 9--Summary of pulse velocity of test specimens before and after exposure to various temperatures.

44.9	45.2	48.6	41.9	39.6	46.7	68.0	69.4	73.8	61.5	63.8	69.7	:	:	:	:	:	:	
2582	2656	2556	2720	2966	2667	1497	1493	1294	1797	1784	1518	:	:	÷	:	:	÷	
4682	4850	4968	4682	4908	5005	4682	4873	4944	4661	4932	5017	:	:	÷	:	:	:	
34.4	34.2	37.4	30.3	36.4	35.0	53.0	52.7	55.6	48.2	51.2	52.9	67.8	69.2	72.4	66.7	66.2	72.2	
3088	3200	3112	3257	3121	3261	2192	2312	2178	2422	2413	2357	1504	1496	1378	1572	1656	1399	
4707	4861	4968	4671	4920	4017	4661	4885	4908	4671	4944	5005	4671	4861	4993	4715	4896	5030	
31.1	31.8	37.3	28.5	25.6	31.9	53.1	51.4	58.0	48.3	46.7	52.8	62.7	63.0	67.6	60.7	60.4	64.7	
3241	3326	3121	3353	3668	3410	2199	2382	2063	2413	2636	2352	1761	1797	1593	1849	1943	1765	
4704	4873	4980	4693	4932	5005	4693	4896	4908	4671	4944	4980	4715	4850	4920	4704	4908	5005	
0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	
1	7	e	4	S	9	-	2	e	4	S	9	-	7	Э	4	S	9	
limestone			dolostone			limestone			dolostone			limestone			dolostone			
300						450						009						

" Each test result is the average of three test results.

				,	Fundan	rental Longitu	dinal Frequ	lency of 1	02 by 203-mm	Cylinders, e	cycles/s"	
Exposure Temper-	Type		W/C Ratio	48-h	Heat Exp	osure	1-Mont	th Heat E	xposure	4-Mont	th Heat E	kposure
ature, °C	of Concrete	MIX No.	(by weight)	Before	After	% Change	Before	After	% Change	Before	After	% Change
75	limestone	-	0.60	9 400	8767	6.7	9433	8600	8.8	9467	8250	12.9
		2	0.45	9 800	9200	6.1	9833	8883	9.7	9800	8433	14.0
		3	0.33	9 750	9400	3.6	9800	8700	11.2	9917	8083	18.5
	dolostone	4	0.60	9 566	9017	5.7	9750	8900	8.7	9767	8433	13.7
		ŝ	0.45	10 000	9467	5.3	9800	0006	8.2	9482	8833	10.3
		9	0.33	10 083	0096	4.8	9867	9050	8.3	9817	8333	15.1
150	limestone	1	0.60	9 450	7833	17.1	9567	2000	17.4	9467	7617	19.5
		2	0.45	9 800	8117	17.2	9808	8033	18.1	9833	7800	20.7
		e	0.33	9 500	7783	18.1	9842	7667	22.1	9933	7417	25.3
	dolostone	4	09.0	9 666	8167	15.5	9767	8017	17.9	:	:	:
		ŝ	0.45	10 067	8667	13.9	9850	8217	16.6		:	:
		9	0.33	10 067	8317	17.4	9783	7800	20.3			

TABLE 10-Summary of fundamental longitudinal frequency of test specimens before and after exposure to various temperatures.

45.6	47.3	52.5	42.2	39.6	47.1	60.2	64.1	66.3	63.9	64.1	68.1	:	:	:	:	:	:	
5133	5167	4717	5633	5917	5200	3733	3517	3333	3517	3533	3133	:	:	:	:		:	
9433	9800	9933	9750	9800	9833	9383	9800	0066	9750	9833	9817	:	:	÷	:	÷		
35.6	35.9	40.9	33.2	30.0	36.7	51.9	51.8	57.6	49.2	50.9	46.3	62.3	67.8	70.9	67.6	65.3	72.6	
6100	6280	5833	6517	6883	6250	4530	4730	4210	4950	4833	5267	3567	3150	2883	3167	3417	2700	
9467	9800	9867	9750	9833	9867	9416	9808	9917	9750	9850	9800	9467	9783	0066	9783	9833	9867	
32.9	33.2	39.3	29.9	28.1	35.1	52.6	51.9	58.7	48.3	46.9	53.9	62.7	63.2	68.0	61.7	61.0	67.0	
6350	6550	5867	6750	7200	6583	4507	4720	4023	4967	5343	4667	3550	3608	3017	3700	3900	3333	
9 467	9 800	9 667	9 633	10 017	10 150	9 500	9 808	9 750	009 6	10 053	10 133	9 517	9 800	9 417	9 666	10 000	10 100	
0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	0.60	0.45	0.33	
-	7	3	4	S	9	-	7	°,	4	S	9	1	7	3	4	S	9	
limestone			dolostone			limestone			dolostone			limestone			dolostone			
300						450						009						

<sup>a</sup> Each value is the average of three test results.

perature has been reported by various investigators, which undoubtedly reflects the considerable influence of different curing or exposure conditions [3].

# Exposure at 150°C

At 150°C, the two types of concrete were found to behave differently under sustained exposure, confirming some of the findings of the first phase of the study.

For the limestone concrete, the reductions in strength after 48 h of exposure were generally comparable to those recorded at  $75^{\circ}$ C, despite the loss of some combined water. The percentage reductions were found to increase slightly with each stage of exposure, reaching a value between 20 and 30% after four months (Table 7). However, there was no visual sign of deterioration of any specimen apart from some slight change in the color of the surfaces (Fig. 7). The slight increase in the weight of the specimens observed over the later stages of the exposure was probably due to some carbonation.

For the dolostone concrete, the effects of exposures of up to one month were similar to those for the limestone concrete, although the strength reductions were somewhat smaller. After four months, however, the results were quite different, as most of the specimens, regardless of the water-to-cement ratio were found to exhibit extensive cracking or complete disruption caused by the instability of some of the aggregate particles (Fig. 8). This particular behavior had also been observed with the same aggregate in the first phase of the study and had subsequently been explained in the following terms [2]:

Petrographic and other related investigations of the rock have indicated that the primary cause of deterioration of some particles is the oxidation of iron sulphide. In this aggregate, fine pyrite is intergranular with dolomite, calcite and clastic material, particularly in shaly sections. Analyses of disintegrated particles from exposed samples revealed that the granular powders consisted of unaltered carbonate and clastic grains, relict pyrite, and sub-micron material containing hygroscopic, iron-rich sulphate. It is concluded that the pyrite had oxidized to iron sulphate hydrate under these particular conditions; the resulting volume change disintegrated the permeable, often weakly bonded particles and stresses arising from aggregate expansion ruptured the concrete. Tests revealed also that the aggregate was relatively stable at 75°C and 300°C and in dry oxygen which suggests that water vapor in a particular pressure-temperature range is critical for the reaction. Water released from the aggregate by sustained heating is in part responsible.

The aggregate particles referred to as unstable were estimated to comprise about 25% of the total aggregate particles.











# Exposure at 300°C

At 300°C, the strength results show no evidence of long-term instability of the dolostone aggregate, as observed at  $150^{\circ}$ C. This is in agreement with what had been observed in the previous phase. For both types of concrete, however, the loss of compressive strength was quite substantial as a result of considerable dehydration of the cement paste. Once again, the percentage of strength loss tended to increase with longer exposures. For example, at 48 h, it was on the order of 30 to 40%, while it increased to more than 50% in most cases after four months (Table 7). Visual examination of the specimens did not reveal any sign of major distress at any stage of the exposure, regardless of the aggregate used (Fig. 9). The gains in weight observed during the later stages of the exposure were probably the result of some carbonation of the concrete.

#### Exposure at $450^{\circ}C$

At  $450^{\circ}$ C, regardless of the concrete composition, the weight loss of the specimens after 48 h of exposure was found to exceed slightly a value equivalent to the estimated total water content of the concrete before exposure (Table 8). This suggests a complete dehydration of the main cementitious phases at this stage, although part of the recorded weight loss could also be accounted for by the decomposition of some organic matter which might have been present in the aggregates. For the limestone concrete, the exposure of up to four months resulted in slight gain in weight, similar to that observed at lower temperatures. For the dolostone concrete, however, an opposite trend was detected at one month, and at four months, drastic weight losses were observed, clearly indicating some decomposition of the aggregate at that stage (Table 8).

As for the strength loss at this temperature, this was on the order of 45 to 55% at 48 h and of more than 65 to 75% at four months, irrespective of the type of aggregate and W/C ratio (Table 7). For the dolostone concrete, the loss at four months was little affected by the decomposition of the aggregate, because of the relatively weak state of the cement mortar at that time. Despite the severe strength loss, the specimens did not exhibit any outward deterioration, apart from the development of some very fine map-cracking at the surface (Fig. 10).

# Exposure at 600°C

Because of the very low residual strength resulting from the exposure at this temperature, the tests were discontinued after one month. By this time all the specimens, though still free of any major surface damage (Fig. 11), had lost more than 75 to 80% of their initial strength.

At 48 h, the decomposition of the dolostone aggregate was again evident, as indicated by large reductions in the weight of the specimens upon exposure.



FIG. 9–Dolostone concrete test specimens after four months of exposure to 300°C sustained temperature. Note: The test specimens do not show the type of degradation noticed after exposure to 150°C.



FIG. 10–Limestone concrete test specimens after four months of exposure to 450°C sustained temperature.



FIG. 11-A dolostone concrete test specimen after one month of exposure to  $600^{\circ}C$  sustained temperature.

For the limestone concrete, there was no sign of such decomposition, although the weight loss observed between 48h and one month might suggest that the limestone aggregate had then reached its limit of stability. As observed at  $450^{\circ}$ C, the performance of the aggregate at this temperature had little effect on the residual strength of the concrete, because of the relatively weak state of the cement mortar.

# Effect of Duration of Exposure

The effect of the duration of exposure on compressive strength is illustrated in Fig. 12, where the average residual strengths of all the concretes (except dolostone at 150°C) are plotted against time of exposure for each particular temperature. Only average values are plotted, as the pattern was found to be mostly independent of the type of aggregate and water/cement ratio.



FIG. 12—Effect of duration of exposure on compressive strength of concrete exposed to various temperatures. Note: The strength data shown are the average values for both limestone and dolostone concretes except for 150°C, in which case the data are for the limestone concretes only.



FIG. 13—Effect of mix proportions on compressive strength of concrete after one month of exposure to various temperatures. Note: The strength data shown are the average values for both limestone and dolostone concretes.



FIG. 14—Relationship between pulse velocity and resonant frequency of concrete exposed to temperatures up to  $600^{\circ}C$ . Note: The data cover both limestone and dolostone concretes.

At 75°C, an increase in length of exposure from 48 h to one or four months had no adverse effect on the compressive strength of the concrete. However, at temperatures of  $150^{\circ}$ C and higher, such increase in exposure resulted in further reduction in strength, the rate of which increased slightly with increasing temperatures. Most of the strength loss occurred during the early stages, that is, within the first month if not during the first 48 h of the exposure. For example, at  $450^{\circ}$ C, about 70% of the total strength loss observed at four months was recorded at 48 h and more than 85% within one month.

#### Effect of Mix Proportions

There was evidence of the effect of the mix proportions on the compressive strength of concrete after exposure to sustained temperatures. The strength losses at high temperatures were consistently smaller for the lean concrete with a water/cement ratio of 0.60 than for concrete with a water/cement ratio of 0.45 or 0.33 (Fig. 13). This was true regardless of the temperature or duration of exposure.

# Pulse Velocity and Resonant Frequency Measurements

Regardless of the concrete composition and condition of exposure, the pulse velocity and resonant frequency of the specimens were affected in the


FIG. 15—Relationship between pulse velocity and compressive strength of concrete exposed to temperatures up to  $600^{\circ}$ C. Note: The data cover both limestone and dolostone concretes.

same manner by the high temperature exposures (Fig. 14). In general, changes in these properties were found to reflect closely changes in the compressive strength of the specimens during exposure. The relationship between pulse velocity and compressive strength is seen to be mostly independent of the type of concrete and condition of exposure (Fig. 15). The only exception was for  $75^{\circ}$ C, in which case the pulse velocity was apparently still affected by the presence of some free moisture.

## Conclusion

1. The dolostone aggregate investigated was found to be unstable under a sustained temperature of  $150^{\circ}$ C, confirming the results of a previous investigation. Such instability is again attributed to the slow oxidation of the pyrite contained in some of the aggregate particles. The resulting expansion causes disintegration of the aggregate and rupture of the concrete. Under similar exposure, concrete made with a limestone aggregate was found to be unaffected.

2. Except for this case, the reductions in the compressive strength of the

specimens under exposure were, in general, independent of the type of aggregate and proportional to the temperature. After 48 h of exposure, the percentage of strength loss ranged from about 20% at 75°C to more than 60% at 600°C. At temperatures of 150°C and higher, an increase in the length of exposure from 48 h to four months resulted in further decrease in strength. In all cases, the major strength loss was still found to occur within the first month of exposure.

3. Regardless of the temperature and duration of exposure, the leaner concrete (W/C = 0.60) was slightly less affected in terms of relative strength loss after exposure than the richer concrete (W/C = 0.45 and 0.33).

4. Both pulse velocity and resonant frequency measurements showed a strong correlation with the compressive strength of the test specimens after exposure. The relationship appears to be little influenced by the composition of the concrete or the condition of exposure.

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# Effect of Temperature and Delivery Time on Concrete Proportions

**REFERENCE:** Gaynor, R. D., Meininger, R. C., and Khan, T. S., "Effect of Temperature and Delivery Time on Concrete Proportions," *Temperature Effects on Concrete*, *ASTM STP 858*, T. R. Naik, Ed., American Society for Testing and Materials, Philadelphia, 1985, pp. 68-87.

**ABSTRACT:** The literature demonstrates that concrete produced at high temperatures [35 versus  $18^{\circ}C$  (95 versus  $65^{\circ}F$ )] or mixed and agitated for 90 versus 20 min, tends to have increased mixing water requirements and reduced strength. The research reported here demonstrates that the additional cement required to compensate for this strength loss can be very modest.

Concrete was mixed at temperatures of 18 and  $35^{\circ}C$  (65 and  $95^{\circ}F$ ). Two series of mixtures were proportioned to produce strengths of 28 to 31 MPa (4000 to 4500 psi) and 34 to 38 MPa (5000 to 5500 psi), utilizing three different material combinations: cement without admixture, cement plus water reducer, and cement plus fly ash. Two cement sources were used and concrete delivery times of 20 or 90 min were simulated. The concrete was retempered to maintain slump. To simulate nonstandard hot weather initial curing, extra cylinders were cured at  $38^{\circ}C$  (100°F) for the first 24 h.

The main considerations were the effect of the research variables on the required cement content to produce a given strength level, on the required mixing water content to maintain a target slump, and on drying shrinkage. The results show that, with one exception, the higher mixing temperatures and extended delivery time had little effect on the concrete properties. One of the two cements used tended to have more strength loss in hot weather when it was used without admixtures. However, when that cement was used with fly ash or a water-reducing admixture, rapid loss of slump and strength was avoided.

An increase in the concrete mixing temperature from 18 to  $35^{\circ}$ C (65 to  $95^{\circ}$ F) required an average increase of 4.7 kg/m<sup>3</sup> (8 lb/yd<sup>3</sup>) of cement to maintain the strength level. An increase in the delivery time from 20 to 90 min required an additional 13.6 kg/m<sup>3</sup> (23 lb/yd<sup>3</sup>) of cement, on the average. The water required to produce the target slump at the higher temperature increased by 2 to 6%. The extended delivery period affected the water requirements by 3 to 13%, and when the extended period was coupled with the higher temperature, the increase in mixing water ranged from 7 to 18%. The hot first-day curing period had a significant effect on the strength, causing a strength loss of about 10%. The variables appeared to have little effect on the drying shrinkage of the concrete.

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**KEY WORDS:** admixtures, cement, compressive strength, concrete, curing, delivery time, drying shrinkage, fly ash, hot weather, shrinkage proportioning, slump loss, temperature, water reducer

There has been much discussion and debate about whether a temperature limit need be applied to concrete produced in hot weather. One argument is that more mixing water is needed for hot concrete, thus reducing the strength and quality for a fixed cement content mixture. The other side is that the production of cooled concrete can be expensive if ice or refrigeration must be used. Appropriate adjustments or changes in the mixture can usually be made to produce concrete of the required quality in the 32 to  $38^{\circ}C$  (90 to  $100^{\circ}F)^2$  temperature range, and in many cases, these changes will be more cost-effective than cooling the concrete.

This discussion is not intended to discourage the use of practical, low-cost measures to keep the concrete temperature down, such as sprinkling or shading aggregate stockpiles or using cooler well water, if available. It is not intended to apply to truly massive concrete members, for which structural integrity considerations may dictate a much lower concrete temperature in order to minimize the amount of early heat generation which must be dissipated from the structure. More typically, upper temperature limits are encountered year-round in hot climate areas or in moderate climate areas in the summer. This research is intended to apply to typical concrete construction for buildings, bridges, pavements, and other applications which are not considered mass concrete, in negligible or moderate exposure conditions.

# Past Research—A Brief Review

Committee 305 of the American Concrete Institute (ACI) has prepared a Report on Hot Weather Concreting [1] which summarizes the technology and includes an extensive list of references. The report includes many of the general problems of hot weather often cited, such as the increase in mixing water demand, the increased rate of slump loss and setting, problems with pumping concrete, the lower strength of concrete in hot weather, and the increased drying shrinkage due to an increase in the water content of concrete. Many of these effects have been verified by investigators, but the magnitude of the change in concrete properties can vary considerably, depending on the characteristics of the materials used, and, as ACI 305 points out, the quality of the curing after placement is very important. Report 305 states that it is impractical to recommend a maximum limiting temperature because circumstances vary widely. It suggests that trial batches should be made at the highest expected temperature for the project to evaluate the hot weather performance of a concrete mixture. Smith [2] also suggests making mixture de-

<sup>&</sup>lt;sup>2</sup>All original measurements were taken in English units.

signs at temperatures of  $32^{\circ}C+$  (90°F+) when temperatures that high are expected.

Two important project-related factors are involved in the research reported here: (1) the effect of the concrete temperature and (2) the effect of the delivery time for the concrete. Klieger [3], Bloem [4], the Bureau of Reclamation [5], and Shalon [6] have reported research on the effect of the concrete temperature. This research shows that, for some of the conditions, where curing after placement was favorable and prolonged mixing was not involved, the strength disadvantage in increasing the concrete temperature from 23 to 38°C (73 to 100°F) was less than 10%. This is an amount which could be compensated for by adjusting the mixture proportions. In a few cases, a particular combination of materials may cause undesirable slump and strength loss problems in hot weather. Examples are reported by Meyer and Perenchio [7], Hersey [8,9], Young [10], and Erlin and Hime [11]. In many of these cases involving slump or strength loss, or both, the early rejection of the tricalcium aluminate  $(3CaO \cdot Al_2O_3)$  (C<sub>3</sub>A) phase of the cement is not properly controlled, that is, the gypsum content is not sufficient to control the rate of the  $C_3A$  hydration. This may be caused by inadequacies in the form or amount of the sulfate in the cement for hot weather performance or an interaction of a water-reducing or retarding admixture, which may upset the balance of soluble sulfate immediately after mixing so as actually to increase the reactivity of the cement, causing increased mixing water demand or early rapid slump loss. Obviously, when these conditions are encountered, a change in materials, proportions, or procedures is needed to eliminate or control the effect.

Research on the effect of longer mixing and delivery times for concrete has been reported by Gaynor [12], Ravina [13], Meininger [14], and Previte [15]. Gaynor employed extended mixing in a small laboratory mixer which was more severe than agitation in a typical ready-mixed concrete truck. In that case, 32°C (90°F) concrete lost slump faster than 21°C (70°F) concrete, requiring about a 50% increase in retempering water to restore slump. Meininger reported on full-scale studies in truck mixers in which there was some delay between batching and mixing and in which the drum was agitated only after mixing was complete. Strength reduction due to needed retempering at 90-min delivery time was about 7% for 18°C (65°F) concrete and 15% for 34°C (93°F) concrete. Previte mixed and agitated concrete for 2 h at 21 and 29°C (70 and 85°F). Concrete with water-reducing admixtures had a slightly higher loss of slump than concrete without, and it was found that one brand of cement did have a tendency for early slump loss. In that case, the admixtures did not reduce the early hydration reactions. Ravina found that loss of slump was higher in concrete with admixtures and that the waterreducing effect was weakened by prolonged mixing and agitation. Tuthill [16] and Daugherty [17] also show data which indicate the reduced effectiveness of water-reducing admixtures at higher temperatures with prolonged mixing.

### **Description of Materials and Tests**

The research reported herein was conducted at the Joint Research Laboratory of the National Sand and Gravel Association and National Ready Mixed Concrete Association (NSGA-NRMCA) located in College Park, Maryland. The principal variables in the study were the temperature of the fresh concrete and the length of time before discharge of the concrete. Concrete was mixed at temperatures of 18 and 35°C (65 and 95°F), and delivery times of 20 and 90 min were simulated in the laboratory for both temperature conditions.

Non-air-entrained 0.034 m<sup>3</sup> (1.2 ft<sup>3</sup>) batches of concrete were mixed in a 0.07-m<sup>3</sup> (2.5-ft<sup>3</sup>) revolving drum mixer for an initial 6-min period. Thereafter, agitation was simulated by intermittent periods of mixing; the drum was stopped between these mixing periods. Prior to discharge, water was added in a remixing cycle to restore the slump to within  $\pm 25 \text{ mm} (\pm 1 \text{ in.})$  of the 100-mm (4-in.) target slump. The desired concrete temperatures were achieved by mixing in controlled temperature areas and by using preconditioned materials and controlled mix water temperature. The mouth of the mixer was covered as much as possible to prevent evaporation.

Other variables include two cements, a Type A lignosulfonate waterreducing admixture, and a Class F fly ash. Cement No. 1 was selected because of a history of slump loss problems in hot weather, whereas Cement No. 2 had not shown such tendencies. Concretes were made using each cement source with (1) no admixtures, (2) the water reducer, and (3) the fly ash. The dosage of the water reducer was 3.3 mL/kg (5 oz/100 lb) of cement for the 18°C (65°F) concrete and 4.6 mL/kg (7 oz/100 lb) of cement for the 35°C (95°F) concrete. Fly ash was used at 20% by weight of the cement plus fly ash.

Concretes were proportioned for two strength levels: 28 to 31 MPa (4000 to 4500 psi) and 34 to 38 MPa (5000 to 5500 psi). The cement content for these mixtures was adjusted on the basis of the anticipated effect of the different test conditions to produce 28-day compressive strengths near these ranges. Later, the mixtures above and below 31 MPa (4500 psi) were interpolated to arrive at the required cement content for a 31-MPa (4500-psi) average strength for each test condition.

The aggregates used were laboratory stock siliceous 19-mm (<sup>3</sup>/<sub>4</sub>-in.) gravel and natural sand with a fineness modulus of 2.83. Two Type I portland cements were used. The compressive strengths of the cements in the ASTM Test for Compressive Strength of Hydraulic Cement Mortar (Using 2-in. or 50-mm Cube Specimens) (C 109-80), were as follows:

Cement No. 1-32.44 MPa (4705 psi) at 7 days; 39.82 MPa (5775 psi) at 28 days; and

Cement No. 2–28.75 MPa (4170 psi) at 7 days; 37.57 MPa (5450 psi) at 28 days.

The two cements were also tested for temperature rise when mixed with water and placed in an insulated container. Cement No. 1 showed significantly more heat generation than Cement No. 2 in the 0 to 30-min time period and also in the 3 to 7-h time period.

After the concrete was discharged at 20 or 90 min, slump and unit weight tests were made and four 152 by 305-mm (6 by 12-in.) cylinders were molded in plastic molds from each batch. Also, one 100 by 355-mm (4 by 14-in.) cylinder was made from each high-strength concrete batch for drying shrinkage tests. Two of the 152 by 305-mm (6 by 12-in.) cylinders and the 100 by 355-mm (4 by 14-in.) cylinder were molded and stored in  $21^{\circ}C$  ( $70^{\circ}F$ ) air for 16 to 20 h prior to being placed in a standard moist room at  $23^{\circ}C$  ( $73^{\circ}F$ ). The other two 152 by 305-mm (6 by 12-in.) cylinders were molded and stored in  $38^{\circ}C$  ( $100^{\circ}F$ ) air for the initial 16 to 20 h, prior to standard moist curing. This was intended to simulate poor initial field curing during hot weather. All the cylinders were protected from loss of moisture during this period by covering them with polyethylene bags, which were sealed with rubber bands.

All the cylinders were removed from the molds at 16 to 20 h and placed in the standard moist room at  $23^{\circ}$ C ( $73^{\circ}$ F), with free moisture maintained on all their surfaces until they were tested at an age of 28 days. The drying shrinkage cylinders were stripped after 1 day and stored in a water tank at  $23^{\circ}$ C ( $73^{\circ}$ F) for 56 days, after which they were allowed to dry in a  $21^{\circ}$ C ( $70^{\circ}$ F) room controlled at  $55 \pm 5\%$  relative humidity. Length change measurements were taken after 28, 91, 180, and 365 days of drying.

## **Presentation of Data**

The properties of the concrete are given in Tables 1 and 2 for the two cements, respectively. The values are averages of replicate batches. The cylinder strength data are based on pairs of cylinders tested for each curing condition from each batch. The medium-strength level concrete is shown in the top half of each table, and the high-strength level in the lower part. The cement and mixing water contents used, based on the actual measured unit weight of the concrete, are shown in Columns 3 and 5. The amount of water-reducing admixture or fly ash used is given in Column 4. The two right-hand columns show the 28-day compressive strength for standard curing in one case and for the 1 day of nonstandard curing at  $38^{\circ}C$  ( $100^{\circ}F$ ) in the other.

Detailed data for each batch are available on request from NSGA-NRMCA [18]. The agreement between replicate batches mixed on different days is quite good. The batch-to-batch standard deviation of strength is 0.85 MPa (123 psi), and the coefficient of variation is 2.7%. Only six batches are outside the target slump range of 75 to 125 mm (3 to 5 in.). For the 99 batches that were mixed, the slump averaged 99 mm (3.9 in.), with a standard deviation of 17 mm (0.7 in.). The batch-to-batch standard deviation for mixing

water content is 2.5 kg/m<sup>3</sup> (4.2 lb/yd<sup>3</sup>), and the coefficient of variation is 1.5%.

Mixing water requirements are given in Table 3. The values are an average for both the medium-strength and high-strength levels since, within the range of cement contents used, the change in proportions did not have much effect on the mixing water needed to obtain the target slump. In fact, in many cases, the higher-strength level required slightly less mixing water than the medium-strength level. Only in two cases did the difference in mixing water requirement between the two strength levels exceed 6 kg/m<sup>3</sup> (10 lb/yd<sup>3</sup>).

The average one-year drying shrinkage data are given in Table 3. Drying shrinkage specimens were made only from the high-strength-level concrete batches. Past research has shown that changes in cement content in the limited range used in this study should not have an important effect on the drying shrinkage. Therefore, the results in Table 3 represent the average for two drying shrinkage specimens made from different batches. The detailed drying shrinkage data for each cylinder is also available on request from NSGA-NRMCA. The variability between drying shrinkage specimens is low. The standard deviation of replicate one-year drying shrinkage results was 0.002 to 0.004% shrinkage. This represents a coefficient of variation of 3 to 6%.

#### **Interpolated Cement Content**

The results of the 28-day, standard-cured cylinder strengths for the medium-strength and high-strength conditions were used to calculate, by straightline interpolation, the cement content required to produce concrete which would *average* about 31 MPa (4500 psi) in compressive strength. These interpolated cement contents are shown in Table 4. The results are shown separately for each cement source. Figure 1 shows an example of the interpolation for the 20-min delivery time. It should be kept in mind that the water-reducing admixture was used at different rates for the two temperature conditions and that fly ash was used as 20% of the cement-fly ash mixture.

These mixtures should average 31 MPa (4500 psi) if standard test methods are employed. For example, they may be suitable for a 28-MPa (4000-psi) specified strength if the proper record of past performance is available. However, if nonstandard testing procedures are employed, particularly in hot weather, lower strengths can be expected and higher cement contents would be required to meet overdesign requirements. The strength results for  $38^{\circ}$ C (100°F) curing for the first day are shown in Tables 1 and 2, and the percentage reduction in strength is presented in Table 5.

Table 6 gives the required cement contents from Table 4 expressed as a percentage of the cement content required for a delivery time of 20-min at  $18^{\circ}$ C (65°F). Also, shown in parentheses, is the incremental increase in cement content in pounds per cubic yard. For example, for Cement No. 1

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Delivery Time, min antno admixtur	Concrete Temperature, °F 6 65 95	Cement Content, Ib/yd <sup>3</sup> A87 514	Amount of Water Reducer, oz/100 lb, or Fly Ash, lb <sup>b</sup> AEDIUM-STRENGTH LEVE	Mixing Water, <sup>c</sup> Ib/yd <sup>3</sup> 1b/302 316	28-Day C Streng Standard 4330 4370	ompressive tth, psi <sup>d</sup> 1 Day at 100°F 3750 3750
	65 95	482 509	:::	324 342	4045 4130	3640 3670
nt + water redu	er 65 65	434 433 429	N 7 N	276 278 306	4115 4265 3670	3615 3800 3300
ıt + fly ash	8 888 8	427 414 430 414	7 104 103	321 301 305 305	3690 4090 4020 4090	3245 3560 3835 3620

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<b>5TH LEV</b>	
-STRENC	
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Cement-no admixture						
20	65	582	:	299	5195	4560
	95	615	:	309	5025	4565
90	65	580		314	5090	4765
	95	609		334	4775	4425
Cement + water reducer						
20	65	533	S	272	5160	4525
	95	568	7	278	5755	5040
90	65	527	5	299	4790	4170
	95	557	7	313	5140	4495
Cement + fly ash						
20	65	522	130	291	5295	4615
	95	538	135	307	5145	4480
90	65	520	130	304	5060	4620
	95	535	134	319	4985	4330
<sup>a</sup> The values are averages o 2% for the cement-only bate	of two or three repl thes, 2 to 4% for the	licate batches mixed he batches with wat	l on different days. <i>A</i> er reducer, and 0.5 t	VII are non-air-entrai	ned concrete. Air con vith fly ash. Detailed	ntents were about 1 to data are available on

 $^{6}$ The amount of water reducer is given in ounces per 100 lb (0.65 mL/kg) of cement; fly ash quantities are given in pounds per cubic yard; fly ash was MFa. request from NSGA-NKMCA. SI units: deg Celstus = (deg Fahrenheit -32)/1.8; 1 lb/yd<sup>3</sup> = 0.5933 kg/m<sup>3</sup>; 1 psi = 6.8948  $\times$  10<sup>-1</sup>

used by weight at the rate of 20% of the total weight of cement plus fly ash. <sup>c</sup>The water required to produce a slump of 4 ± 1 in. (100 ± 25 mm) at the time of discharge.

 $^{d}$ Standard curing is 21°C (70°F) for the first 16 to 20 h followed by curing in a standard moist room at 23°C (73°F); one day at 38°C (100°F) curing is 30°C (100°F) for 16 to 20 h followed by curing in the standard moist room.

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Simulated	Concrete	Cement	Amount of Water Reducer.	Mixine	28-Day ( Stren	Compressive eth. psi <sup>d</sup>
Delivery Time, min	Temperature, °F	Content, Ib/yd <sup>3</sup>	$oz/100$ lb, or Fly Ash, $lb^b$	Water, <sup>c</sup> lb/yd <sup>3</sup>	Standard	1 Day at 100°F
		2	fedium-Strength Leve			
Cement-no admixtur	ų					
20	65	487		304	4290	3800
	95	520	•	309	4700	4280
6	65	484	•	316	4215	3915
	95	517		337	4425	4180
Cement + water reduc	cer					
20	65	434	ŝ	273	4160	3870
	95	434	7	284	4135	3820
90	65	431	S	305	3650	3415
	95	428	7	318	3725	3340
Cement + fly ash						
20	65	416	104	288	4090	3800
	95	432	108	305	4140	3785
66	65	414	104	301	3965	3680
	95	431	108	325	4110	3735

TABLE 2—Properties of laboratory-mixed concrete for Cement No. 2 (Series 211)."

		H	ligh-Strength Leve	L		
Cement-no admixture			÷			
20	65	586	:	292	5565	5080
	95	618	:	305	5730	5285
6	65	582	:	304	5445	5020
	95	612	:	330	5540	5175
Cement + water reducer						
20	65	536	5	261	5715	5195
	<del>95</del>	570	7	283	5970	5370
06	65	529	5	299	4915	4570
	95	560	7	313	5460	5025
Cement + fly ash						
20	65	524	131	284	5800	5090
	95	542	136	301	5735	5125
90	65	523	130	301	5315	4735
	95	540	136	320	5525	5020
<sup>a</sup> The values are average 2% for the cement-only bir request from NSGA-NRA <sup>b</sup> The amount of water to used by weight at the rate <sup>c</sup> The water required to <sup>d</sup> Standard curing is 21°d 38°C (100°F) for 16 to 20	s of two or three atches, 2 to $4\%$ f ACA. SI units: d ACA. SI units: d aducer is given in of 20% of the to produce a slump C (70°F) for the f h followed by cu	replicate batches mix replicate batches with w eg Celsius = (deg Fa ounces per 100 lb (0.1 tal weight of cement of $4 \pm 1$ in: (100 $\pm 1$ irst 16 to 20 h follower ring in the standard	ted on different days. ater reducer, and $0.5$ threnheit $- 32)/1.8$ ; 0.5 mL/kg) of cement; plus fly ash. 25 mm) at time of di d by curing in a stands moist room.	All are non-air-entrai to $1.5\%$ for batches v $1 \text{ lb/yd}^3 = 0.5933 \text{ kg}$ fly ash quantities are scharge. red moist room at 23°	ned concrete. Air conwith fly ash. Detailed $y'm^3$ ; 1 psi = 6.894, given in pounds per 6 (73°F); one day at 5	titents were about 1 to data are available on $8 \times 10^{-3}$ MPa. :ubic yard; fly ash was $8^{\circ}$ C (100°F) curing is

			Cement No. 1			Cement No. 2	
Simulated Delivery Tı Time. min	Concrete emperature, ∘F	Average Mixing Water, Ib/vd <sup>3b</sup>	Mixing Water Increase, Ib/vd <sup>3c</sup>	1-Year Drying Shrinkage, %, d	Average Mixing Water, Ib/yd <sup>3b</sup>	Mixing Water Increase, Ib/yd <sup>3</sup>	1-Year Drying Shrinkage,
Cement-no admixture 20	65	300	:	0.059	298		0.051
06	95 95 95	312 319 338	19 38 38	0.055 0.064 0.060	307 310 333	9 35 35	0.047 0.058 0.052
Average				090.0			0.052
Cement + water reducer 20	65 05	274 278	: •	0.061	267 783	: 4 : 4	0.054
90	65 95 95	303 317	29 43	0.066 0.066	302 316	35 49	0.059
Average				0.064			0.057
Cement + fly ash 20 90	95 95 95	296 306 318	10 8 22	0.051 0.051 0.052 0.049	286 303 301 322	 17 36	0.053 0.048 0.054 0.050
Average				0.051			0.051

	Cement Content, 1b/yd <sup>3</sup> , to Produce 4500 psi at 28 Days (% of Cement Content for 65°F, 20-min Delivery Condition)					
Simulated	Cemen	it No. 1	Cemer	1t No. 2		
Time, min	65°F Concrete	95°F Concrete	65°F Concrete	95°F Concrete		
Cement-no admixture						
20	506	539	503	501		
90	525	566	507	523		
Cement + water reducer <sup><math>b</math></sup>						
20	470	454	456	461		
90	502	500	497	487		
Cement + fly $ash^c$						
20	451	443	442	457		
90	459	476	457	461		

 TABLE 4—Interpolated cement content requirement to produce 4500-psi average

 compressive strength (Series 211).<sup>a</sup>

<sup>a</sup>The cement contents are those required to produce a 28-day standard-cured cylinder strength of 4500 psi. They are interpolated from the strength data for the medium and high-strength concretes in Tables 1 and 2. SI units: deg Celsius = (deg Fahrenheit - 32)/1.8; 1 lb/yd<sup>3</sup> = 0.5933 kg/m<sup>3</sup>; 1 psi =  $6.8948 \times 10^{-3}$  MPa.

<sup>b</sup>The 18°C (65°F) concrete contains 3.3 mL/kg (5 oz/100 lb), and the 35°C (90°F) concrete contains 7 oz/100 lb (4.6 mL/kg) of water reducer.

<sup>c</sup>The values given are cement content only. Fly ash is also used at the rate of 20% of the total weight of cement + fly ash, that is, 1 lb of fly ash for each 4 lb of cement.



FIG. 1—Example of interpolation of strength data—20 min delivery time. (SI units:  $65^{\circ}F = 18^{\circ}C$ ;  $95^{\circ}F = 35^{\circ}C$ ; 1 lb/yd<sup>3</sup> = 0.5933 kg/m<sup>3</sup>; 1 psi = 6.8948 × 10<sup>-3</sup> MPa.)

	<b>6</b>	20 m	in, %	90 m	in, %
Condition	No.	65°F	95°F	65°F	95°F
Cement—no	1	13.1	10.5	9.2	9.4
admixture	2	10.9	9.2	7.3	9.6
Cement + fly ash	1	12.9	13.2	9.2	12.7
(80-20)	2	8.7	8.5	9.0	9.1
Cement + water	1	14.4	11.2	12.3	12.4
reducer	2	7.6	8.3	6.9	9.0

TABLE 5-Strength loss, percentage from standard cure for 24-h cure at 100°F.<sup>a</sup>

<sup>*a*</sup>SI units: deg Celsius = (deg Fahrenheit - 32)/1.8.

TABLE 6—Percentage of cement content at 65°F, 20-min delivery condition required to
produce 4500-psi average compressive strength concretes. <sup>a</sup>

	Cement Content, lb/yd <sup>3</sup> , to Produce 4500 psi at 28 Days (as a % Based on 20-min, 65°F Condition) <sup>b</sup>					
Simulated	Cemen	it No. 1	Cemer	nt No. 2		
Time, min	65°F Concrete	95°F Concrete	65°F Concrete	95°F Concrete		
Cement-no admixture						
20	100 ()	107 (33)	100 ()	100(-2)		
90	104 (21)	112 (60)	101 (4)	104 (20)		
Cement + water reducer						
20	100 ( )	97 (-16)	100 ()	101 (5)		
90	107 (32)	106 (30)	109 (41)	107 (31)		
Cement + fly ash						
20	100 ( )	98(-8)	100 ( )	103 (15)		
90	102 (8)	106 (25)	103 (15)	104 (19)		
Overall average of all conditions						
20	100 ( )	101 (3)	100 ()	101 (6)		
90	104 (20)	108 (38)	104 (20)	105 (23)		

<sup>a</sup>SI units: deg Celsius = (deg Fahrenheit - 32)/1.8; 1 psi =  $6.8948 \times 10^{-3}$  MPa.

<sup>b</sup>The incremental increase or decrease in cement content in terms of pounds per cubic yard from the 20 min-65°F ( $18^{\circ}$ C) condition are shown in parentheses.

without admixture, an increase of 7% was needed to maintain the same strength level for a  $35^{\circ}C$  ( $95^{\circ}F$ ) concrete temperature at a delivery time of 20 min. For Cement No. 2, no increase was needed. It is interesting to note that the data in this report indicate that the difference between the two cements is greatly reduced when the water reducer or fly ash is used. The overall average percentages for all conditions are shown in the lower part of Table 6.

# **Discussion of Results**

## **Cement** Content

The increase in cement content needed to compensate for a change in concrete temperature to 35 from  $18^{\circ}$ C (95 from  $65^{\circ}$ F) is modest in most of the cases studied. Tables 4 and 6 show the cement content for each condition, and they are plotted versus delivery time in Fig. 2. The increase averages 2% or 4.7 kg/m<sup>3</sup> (8 lb/yd<sup>3</sup>).

The Cement No. 1 without admixture showed the only large increase in cement content required in going from 18 to  $35^{\circ}C$  (65 to  $95^{\circ}F$ ). This is the cement that was specifically chosen because of its poor hot weather performance. For the 20-min delivery time, the cement content increased from 300 to 320 kg/m<sup>3</sup> (506 to 539 lb/yd<sup>3</sup>), and the increase for the 90-min time was from 311 to 366 kg/m<sup>3</sup> (525 to 566 lb/yd<sup>3</sup>). This effect is shown in the lefthand part of Fig. 2. Note that the cement content line for Cement No. 1 used in  $35^{\circ}C$  ( $95^{\circ}F$ ) concrete without admixture is much higher than that for the other conditions. However, when this same cement was used with a water reducer or with fly ash, the dramatic increase disappeared. In most of the cases shown in Fig. 2, the dashed lines for the  $35^{\circ}C$  ( $95^{\circ}F$ ) concrete with several conditions.

For Cement No. 2, the change in cement content in going from 18 to  $35^{\circ}$ C (65 to  $95^{\circ}$ F) was small for all conditions. The higher temperature necessitated an average increase of 4.1 kg/m<sup>3</sup> (7 lb/yd<sup>3</sup>) for the cement without admix-



FIG. 2—Cement content required for 4500 psi compressive strength (SI units: 4500 psi = 31 MPa;  $65^{\circ}F = 18^{\circ}C$ ,  $95^{\circ}F = 35^{\circ}C$ ;  $1 \text{ lb/yd}^3 = 0.5933 \text{ kg/m}^3$ ).

ture, required no increased cement content when used with the water reducer, and required less than  $6 \text{ kg/m}^3$  (10 lb/yd<sup>3</sup>) with the fly ash.

There is a tendency for the cement requirement to increase as the delivery time is extended to 90 min. This increase averaged 5%, or 14 kg/m<sup>3</sup> (23 lb/yd<sup>3</sup>) (see Table 6). This is an expected result of early cement hydration that causes slump loss and the needed addition of more mixing water to maintain slump. Additional cement should maintain average design strength if extended delivery times are expected. Note, however, from Fig. 2 that the slope of the lines is about the same for concrete at both 18 and  $35^{\circ}C$  (65 and  $95^{\circ}F$ ). This again indicates that there is no drastic strength penalty for the  $35^{\circ}C$  (95°F) concrete as long as the cylinders are tested in accordance with standard conditions.

From a material selection standpoint, the mixtures with water reducer or with fly ash required 23.7 to 29.7 kg/m<sup>3</sup> (40 to 50 lb/yd<sup>3</sup>) less cement for the 20-min delivery than the concrete without admixture (disregarding the hotter concrete with Cement No. 1). Also, the fly ash performed equally well for a 90-min delivery. Since the fly ash was used at a rate of 65 to 77 kg/m<sup>3</sup> (110 to 130 lb/yd<sup>3</sup>), it appears to be about 40% efficient in replacing cement in this particular case, except for the hot concrete with Cement No. 1, in which the fly ash eliminated the strength problem and replaced the cement at an effectiveness approaching 90%.

The cement with water reducer did not do as well at the 90-min delivery time as it did at 20 min. Figure 2 shows that the lines are steeper for that condition. In the longer delivery the water-reduced concrete lost slump faster, requiring more mixing water than the fly ash concrete. Therefore, the cement content needed for 90-min delivery increased by 17.8 to 23.7 kg/m<sup>3</sup> (30 to 40 lb/yd<sup>3</sup>). Other researchers have reported decreased effectiveness of water reducers under similar conditions [13, 15, 16, 17].

# Mixing Water Requirement

Mixing water requirements for the different test conditions were fairly close for batches mixed with Cement No. 1 or Cement No. 2. This can be seen from Table 3, in which the average values for mixing water are compared. Cement No. 1 did average 3 kg/m<sup>3</sup> (5 lb/yd<sup>3</sup>) higher for the no-admixture condition in which, as discussed previously, a much greater cement content was needed to maintain strength for the 35°C (95°F) concrete. However, this modest increase in mixing water is not sufficient to explain the poor strength performance in this particular case.

The increase in mixing water over that needed for the short delivery time at  $18^{\circ}C$  (65°F), given in Table 3, shows that an additional 6.5 kg/m<sup>3</sup> (11 lb/yd<sup>3</sup>) or 3%, of mixing water was needed on the average for the 35°C (95°F) concrete. When the higher temperature and longer mixing time are combined, the increase in mixing water averaged 19.6 kg/m<sup>3</sup> (33 lb/yd<sup>3</sup>), or

10%, for the no-admixture and fly ash conditions and 27.3 kg/m<sup>3</sup> (46 lb/yd<sup>3</sup>), or 16%, for the concrete with water reducer.

In Fig. 3 the mixing water demands for the two cement conditions have been averaged. Therefore, the figure summarizes the effects of temperature and delivery time on the mixing water requirement for the three mixture types. The fly ash concrete consistently required 3 to 6 kg/m<sup>3</sup> (5 to 10 lb/yd<sup>3</sup>) less mixing water than the concrete without admixture, except that the reduction was closer to 12 kg/m<sup>3</sup> (20 lb/yd<sup>3</sup>) for the 90-min concrete with Cement No. 1 (see Table 3). The concrete with water reducer showed at least a 15-kg/m<sup>3</sup> (25-lb/yd<sup>3</sup>) reduction in mixing water for the shorter 20-min delivery time, but its effect was diminished at the 90-min delivery time. This is shown by the steepness in the lines in the right-hand part of Fig. 3. The concrete with water reducer required more water to restore slump at 90 min than the other two mixture conditions.

### Drying Shrinkage

The concrete temperature and mixing water quantity had relatively little effect on the drying shrinkage. The one-year drying shrinkage data are shown in Table 3 and Fig. 4. The most noticeable effects are the tendency for the water-reducer concrete to shrink 5 to 10% more on the average than the concrete without admixture. Cement No. 1 showed a higher shrinkage in all but the fly ash concrete. The fly ash concrete was consistently low in shrinkage.



FIG. 3—Average mixing water content for  $4 \pm 1$ -in. slump. (SI units:  $4 \pm 1$  in. = 100 ± 25 mm; 65°F = 18°C, 95°F = 25°C; 1 lb/yd<sup>3</sup> = 0.5933 kg/m<sup>3</sup>.)



FIG. 4—Drying shrinkage versus mixing water content. (SI units:  $65^{\circ}F = 18^{\circ}C$ ,  $95^{\circ}F = 35^{\circ}C$ ; 1 lb/yd<sup>3</sup> = 0.5933 kg/m<sup>3</sup>.)

Traditionally, in concrete technology it has been stated many times that drying shrinkage is highly dependent on the amount of mixing water used. That is not the case here. Figure 4 shows a graph of one-year drying shrinkage versus mixing water. The various conditions in this study caused the mixing water to range from 154 kg/m<sup>3</sup> (260 lb/yd<sup>3</sup>) to almost 201 kg/m<sup>3</sup> (340 lb/yd<sup>3</sup>). There is no correlation for the data taken as a whole, or for the fly ash concrete or the concrete without admixture. For the water-reduced concretes, which tended to have higher shrinkage anyway, there is some correlation. However, the correlation coefficient between shrinkage and the mixing water requirement is less than 0.6, which indicates correlation at the 95% level of confidence but not at the 99% level. In other studies at the NSGA-NRMCA Joint Research Laboratory [18], only very small increases in drying shrinkage were obtained when the amount of mixing water was increased by modest amounts, such as that necessary to increase slump from 50 to 150 mm (2 to 6 in.).

## Hot 38°C (100°F) Curing for the First Day

Table 5 shows the percentage strength loss for first-day curing at  $38^{\circ}$ C (100°F) versus standard curing. This one-day deviation from the standard cost an average of 11.7% strength loss for those concretes made with Cement No. 1, and 8.7% strength loss for Cement No. 2. To overcome this additional loss of strength, the data in this study show that approximately 26.7 kg/m<sup>3</sup> (45 lb/yd<sup>3</sup>) of additional cement would be required. Therefore, the effect of nonstandard curing of cylinders is more significant than the effect of increased concrete temperature or lengthened delivery time. This gives added emphasis to the importance of following proper cylinder curing procedures in hot weather.

# Effect of Temperature and Delivery Time on Strength

Table 7 demonstrates what would happen if cements were chosen on the basis of experience at cool temperatures and short haul times, and later the temperatures or haul time were increased. A number of interesting trends are noted. Based on overall averages, the effect of extending delivery time from 20 to 90 min is modest for cement and cement-fly ash mixes—a strength loss of 1 to 4%. For mixtures with a water reducer, the effect was more significant with a strength loss of about 10%.

Increasing the temperature from 18 to 35°C (65 to 95°F) has a rather

			Strengt	h, % of 20-r	nin Delivery	at 65°F
	- ·	Cement	20	min	90	min
Condition	Cement No.	Content, lb/yd <sup>3b</sup>	65°F	95°F	65°F	95°F
Cement—no admixture	1 2	506 503	100 100	95 100	96 99	91 95
Cement + water reducer <sup><math>c</math></sup>	1 2	451 442	100 100	104 98	92 88	93 91
Cement + fly $ash^d$	1 2	470 456	100 100	101 95	98 96	96 94

 

 TABLE 7—Effect of temperature and delivery time on concretes designed to produce 4500 psi when mixed for 20 min at 65°F.<sup>a</sup>

<sup>a</sup>SI units: deg Celsius = (deg Fahrenheit - 32)/1.8; 1 lb/yd<sup>3</sup> = 0.5933 kg/m<sup>3</sup>; 1 psi = 6.8948 × 10<sup>-3</sup> MPa.

<sup>b</sup>Cement content required to produce 31 MPa (4500 psi) for 18°C (65°F) concrete mixed 20 min.

<sup>c</sup>Water reducer dosage is 3.3 mL/kg of cement (5 oz/100 lb) at  $18^{\circ}\text{C}$  (65°F) and 4.6 mL/kg of cement (7 oz/100 lb) at  $35^{\circ}\text{C}$  (95°F).

<sup>d</sup>Cement and fly ash use in ratios of 4:1 by weight.

modest effect on concrete strength. For individual conditions, the strengths ranged from an increase of 4% to a loss of 5%. Here the increased dosage of water reducer used at 35°C (95°F) offsets the temperature effect in that condition. The average strength losses for cement and cement-fly ash were only 3 and 2%, respectively.

# Conclusions

1. A concrete temperature of  $35^{\circ}C$  ( $95^{\circ}F$ ) or a 90-min delivery time had a modest effect on the required cement content. Mixture adjustments can be made to compensate for these effects for the level of strength included in this study.

2. Higher concrete temperatures, with one exception, had little effect on the cement content required to produce concrete averaging 31 MPa (4500 psi) in strength. One cement did have a significant strength loss at  $35^{\circ}$ C ( $95^{\circ}$ F) when used without admixture or fly ash. Otherwise, the increase in concrete temperature from 18 to  $35^{\circ}$ C (65 to  $95^{\circ}$ F) required an average increase of only about 4.7 kg/m<sup>3</sup> (8 lb/yd<sup>3</sup>) of cement.

3. An increase in delivery time from 20 to 90 min required an average additional 13.6 kg/m<sup>3</sup> (23 lb/yd<sup>3</sup>) of cement to maintain the desired strength.

4. The mixing water needed to obtain the required slump increased 2 to 6% for  $35^{\circ}C$  ( $95^{\circ}F$ ) concrete and 3 to 13% when the effects of the hotter temperature were combined with a 90-min delivery time.

5. Use of the Type A water reducer or Class F fly ash reduced the effects of the increased concrete temperature.

6. Use of the Class F fly ash reduced the effects of the extended delivery period.

7. The concretes mixed with the water reducer showed the most slump loss during the extended mixing time.

8. There was no evidence that the increased mixing water or higher temperatures had any detrimental effect on shrinkage.

9. The concrete with fly ash exhibited the lowest one-year drying shrinkage and the water-reduced concretes the highest drying shrinkage.

10. Nonstandard first-day curing at  $38^{\circ}$ C (100°F) had a significant effect on strength. It caused strength losses from 9 to 12% of the strengths of those same concretes when standard cured. This is equivalent to an average increase of 26.7 kg/m<sup>3</sup> (45 lb/yd<sup>3</sup>) of cement to compensate for the hot first-day curing.

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# Effect of Hot Weather Conditions on the Strength Performance of Set-Retarded Field Concrete

**REFERENCE:** Mittelacher, M., "Effect of Hot Weather Conditions on the Strength Performance of Set-Retarded Field Concrete," *Temperature Effects on Concrete, ASTM STP 858*, T. R. Naik, Ed., American Society for Testing and Materials, Philadelphia, 1985, pp. 88-106.

**ABSTRACT:** Field test reports from seven major projects representing 1336 individual tests were evaluated for the effect of hot weather conditions, including the effects of placing temperatures of up to  $37^{\circ}C$  (99°F) on the 28-day compressive strength of set retarded concrete. The information was based on reports from independent agencies testing the concrete in behalf of the owner of the project. For the majority of the projects, the test specimens had been left exposed to ambient conditions during the initial curing period at the site. The concrete was supplied by different producers to power plant, high rise, mass transit, or highway projects in Florida and Atlanta, Georgia. The data were analyzed for temperature-strength relationships and for significant differences in average strength and standard deviation between data groups of concrete placed at temperatures below and above  $32^{\circ}C$  (90°F). The statistical analysis revealed the absence of a significant differences in the average strength and standard deviation of concrete of lower and higher placing temperature. Exceptions to these observations showed a more favorable performance in concrete of higher placing temperature.

**KEY WORDS**: admixtures, cement composition, compressive strength, concrete, correlation analysis, curing, field tests, hot weather, scatter diagram, set retarder, standard deviation, statistical evaluation, temperature

Observations over many years on the performance of set-retarded concrete in hot weather in the Florida-Georgia area appeared to indicate little or no adverse effect on the 28-day strength of ambient or placing temperatures. These observations were in conflict with a fairly solid body of information, from both the field and laboratory investigations, which showed that elevated

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mixing and early curing temperatures caused lower concrete strengths at 28 days. Available information, primarily that developed in laboratory research [1-3], however, indicated that observations of lower strengths resulting from elevated temperatures were made on plain concrete or concrete in which the setting characteristics had not been modified by admixtures.

To determine whether the field observations of a minimal adverse effect of elevated temperatures on the strength of set-retarded concrete were chance occurrences or statistically significant, the data from seven projects, representing a total of 1336 field tests, and a range of placing temperatures from  $10^{\circ}C$  ( $50^{\circ}F$ ) to  $37^{\circ}C$  ( $99^{\circ}F$ ), were evaluated for the effects of temperature on strength. On five of the seven projects, the test specimens were left unprotected from ambient conditions during early site curing. Hot weather conditions that could impair the concrete strength at these sites thus included both high placing temperatures and high early curing temperatures.

## The Test Data and Their Evaluation

The information on concrete temperatures at the time of sampling and on 28-day compressive strengths originated from the reports of independent laboratories testing the concrete in behalf of the owner of the project, and it covered power plant, high rise, urban expressway, and mass transit projects in Florida and Atlanta, Georgia. The specified minimum strengths varied from 23 MPa (3400 psi) to 48 MPa (7000 psi). The test results from the individual projects are presented in the form of scatter diagrams (Figs. 1 through 7), in which the 28-day strengths are plotted over the corresponding temperatures of the specimens. Multiple occurrences of given temperature-strength combinations are shown by the numbers in the appropriate locations. Background information on individual projects and characteristics of the concrete used in them is provided in Table 1.

The data were examined for their temperature-strength correlation. Since some importance is assigned by specifiers to  $32^{\circ}C(90^{\circ}F)$  as a maximum tolerable temperature for hot weather concrete, the data on concrete with placing temperatures of  $32^{\circ}C(90^{\circ}F)$  or lower were compared with the data on concrete of higher placing temperatures.

Conditions of nonstandard initial curing in a hot weather environment were not amenable to statistical evaluation, because the available data did not include early temperature histories of specimens, nor were any control specimens cured under standard conditions. The group of specimens exposed to ambient hot weather conditions is identified later, in the section on Initial Site Curing. The hot weather exposure of this group is seen as a factor that reinforced in a general way the findings of the statistical evaluation of placing temperature effects on strength.





MITTELACHER ON HOT WEATHER EFFECTS ON FIELD CONCRETE

91









10.0

28-DAY COMPRESSIVE STRENGTH, PSI--Y



= 0.05 n = 246



FIG. 5—Compressive strength versus placing temperature of 3500-psi structural concrete in Project E (metro rail. Miami, FL, Supplier L).

## **Concrete Composition**

Each of the concrete mixtures used in the various projects included a different cement-admixture combination. Observations, thus, had a fairly broad base in regard to materials variables of primary importance to strength performance. It can be assumed that the concrete suppliers had selected jobtested materials with a record of producing strength- and cost-efficient concrete with a minimum of handling and performance problems, specifically related to the hot weather conditions prevailing during much of the year in the Florida-Georgia area. Since cement chemistry has a bearing on admixture performance in concrete, typical mill analysis information is provided in Table 2. Cement properties considered advantageous to admixture efficiency include low alkali and low tricalcium aluminate  $(3 \text{ CaO} \cdot \text{Al}_2\text{O}_3)$  (C<sub>3</sub>A) contents. It can be seen that all the cements have alkali contents near or below the 0.60% limit for low-alkali cements of the ASTM Specification for Portland Cement (C 150-83a). The C<sub>3</sub>A contents are also relatively low. In the Florida projects, the coarse aggregate consisted of crushed Florida limestone with an absorption of 2.5 to 6.5%. A high-density granite was used in the Atlanta project. Manufactured sands were included in concrete for the Miami and Atlanta projects. At other Florida locations, natural silica sand of a low fine-



FIG. 6—Compressive strength versus placing temperature of 4000-psi structural concrete at seven days in Project F (high-rise office building, Atlanta, GA).

ness modulus of 2.20 and lower was used. The admixtures complied with the ASTM Specification for Chemical Admixtures for Concrete (C 494-82).

The concrete mixtures were proportioned for structural uses, except for that used on Project B, which was a mass concrete of leaner cement and higher coarse aggregate content. Regarding admixture use, the dosage of the set-retarding admixtures (ASTM Specification C 494-82, Type D) was kept uniform on five of the seven projects, regardless of the ambient temperature conditions. On two of the projects, admixture use was modified as follows: on Project A the dosage of the modified lignin retarder was increased from 3.9 to 5.2 mL/kg cement (from 6 to 8 fl oz/100 lb cement) whenever the prevailing concrete temperatures rose to  $29^{\circ}$ C ( $85^{\circ}$ F) or higher levels. Also at  $29^{\circ}$ C ( $85^{\circ}$ F) and higher concrete temperatures, the set-retarder formulation (ASTM Specification C 494-82, Type D) of the glucose polymer admixtures used on Project C was substituted for the normal set (ASTM Specification C 494-82, Type A) formulation. A naphthalene-formaldehyde superplasticizer (ASTM Specification C 494-82, Type F) was included in the 48-MPa





	TABLE 1	— Project infor	mation and con	crete properties.			
			Project, Ty	pe of Project, a	nd Location		
			ť				
	Α,	B,	Power	Ď	щ	ц,	ۍ ن
	Mass	Power	Plant,	Urban	Mass	Office	Office
	Transit,	Plant,	W. Central	Expressway,	Transit,	Complex,	Complex,
Characteristic <sup>a</sup>	Miami	N. Florida	Florida	Tampa	Miami	Atlanta	Miami
Class of concrete, psi	4000	4000	4000	3400	3500	4000 at	7000
	structural	mass	structural	structural	structural	7 days	structural
Period of use, month/year	4/81 to 12/81	4/80 to 8/80	9/79 to 10/80	1/80 to 12/80	7/81 to 12/81	6/82 to 9/82	7/82 to 1/83
Concrete temperature limit, °F	none	none	66	$95 \pm 2$	none	none	100
Range of placing temperatures, °F	67 to 99	69 to 98	53 to 90	50 to 97	71 to 98	73 to 93	61 to 97
Cement, lb/yd <sup>3</sup>	536	520	555	$564^{b}$	500	677	752
Slump range, in.	2 to 4	1 to 4	1 to 4	1 to 4	3.5 maximum	4 to 8	6 to 8
Range of air content, %	3 to 6	3 to 6	3 to 6	3 to $7^c$	3 to 6	no air	no air
Set-retarding admixture	lignin	lignin	glucose <sup>d</sup>	lignin	HC	glucose	lignin <sup>/</sup>
Admixture dosage, oz/100 lb cement	$6 \text{ or } 8^8$	7.5	4	9	e	e	æ
Number of tests	67	238	300	246	58	104	293
Average 28-day strength, psi	5687	5446	5225	4879	4905	5783	8178
Standard deviation, psi	441	348	472	558"	361	447	476
$^{a}1000 \text{ psi} = 6.895 \text{ MPa}; \text{ degree Celsiu}$	is = (degree Fahr	enheit $-32)/1$	l.8; 100 lb/yd <sup>3</sup> =	= 59.3 kg/m <sup>3</sup> ; 1	in. = 25.4 mm	; 1 fl oz/100 lb	= 0.652 mL/kg.
"Includes 112 lb/yd". Class F tly ash.	•		•				
Air content range was 3 to 6% for str dNormal seat formulation was used at 5	ructural concrete	and 4 to 7% to	r bridge deck co F	ncrete.			
$^{\mu}$ HC = hvdroxvlated carboxvlic acid.							
fin addition to the set retarder a number	htholono-formolds	huda cunaenla	ticizar was nead	at 8 az/100 lb	coment		

<sup>J</sup>In addition to the set retarder, a naphthalene-formaldehyde superplasticizer was used at 8 oz/100 lb cement. <sup>g</sup>The higher dosage was used at concrete temperatures of 85°F and higher. <sup>h</sup>The greater data scatter was probably caused by two ranges of air content required in different portions of the work and by the data covering several contracts handled by different contractors and testing agencies (see Footnote c).

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			Pro	ject and Cement	Type		
	Α, Ι	B, I/II	C, II	D, I	E, I	F, I	G, I
Chemical composition, %							
SiO,	21.8	21.5	23.3	20.1	21.4	21.2	21.5
AŀŐ,	4.6	4.3	4.3	5.6	5.0	5.6	5.2
Fe,O,	3.7	3.3	3.5	3.6	2.8	2.7	2.8
CaÔČ	65.1	62.9	:	63.7	66.1	65.2	65.7
MgO	1.0	3.7	0.5	1.0	1.1	0.6	1.0
sõ	2.7	2.7	2.4	3.4	2.3	3.2	2.4
Na, O equivalent	0.34	0.57	0.32	0.38	0.38	0.40	0.31
Loss on ignition	1.0	0.9	1.5	2.1	1.7	0.9	1.2
Insoluble residue	0.14	0.57	0.18	0.34	0.20	0.17	0.20
C <sub>1</sub> S	56.0	51.3	49.0	55.3	62.1	53.5	58.9
C <sub>3</sub> A	6.1	5.8	5.4	8.6	8.5	10.3	0.6
Physical tests							
Fineness, Blaine, cm <sup>2</sup> /g	3430	3780	3850	3870	3850	3770	3900
Soundness, % expansion	0.02	0.06	0.02	0.04	0.04	0.03	0.04
Gillmore set, initial, h	2:30	3:25	2:40	2:12	3:20	$1:10^{b}$	3:05
Gillmore set, final, h	4:20	5:40	4:25	4:10	5:40	$2:40^{b}$	5:55
Air content, %	7.8	8.7	7.7	10.2	:	7.9	•
Cube strength, psi <sup>a</sup>							
1 day	1840	1970	÷	1950	1780	:	1720
3 days	3280	3510	2740	3550	3370	3130	3520
7 days	4520	4540	3810	4540	4740	4330	4670

<sup>&</sup>lt;sup>a</sup>1000 psi = 6.895 MPa. <sup>b</sup>Vicat time of set.

(7000-psi) concrete on Project G, in addition to the modified lignin set retarder, which was used at standard dosage throughout seasonal temperature changes.

### **Initial Site Curing**

Test specimens were left exposed to ambient conditions during initial site curing on five of the seven projects. Curing on Project C complied with the ASTM Method of Making and Curing Concrete Test Specimens in the Field (ASTM C 31-83), which requires that specimens be maintained at a temperature between 16°C ( $60^{\circ}$ F) and 27°C ( $80^{\circ}$ F) during the initial 24-h curing period after molding. On Project B, a sizable number of specimens made during the frequent high-volume placements remained under ambient conditions because of a limited availability of curing boxes. Following the initial curing period, the specimens from all the projects were stored under the standard curing conditions required for acceptance testing until the time of test.

The temperatures of ambient air shown on the test reports were those recorded at the time of sampling. When recorded early in the day under hot weather conditions, these temperature data did not provide reliable indications of exposure conditions of test cylinders during the remainder of the initial curing period. Maximum air temperatures recorded on the various projects ranged from  $36^{\circ}C$  ( $96^{\circ}F$ ) to  $39^{\circ}C$  ( $102^{\circ}F$ ). No information was available on the peak temperatures attained by unprotected specimens. However, temperatures in excess of  $46^{\circ}C$  ( $115^{\circ}F$ ) were observed in test cylinders exposed to the sun during site curing. The specimens that represented placing temperatures higher than  $32^{\circ}C$  ( $90^{\circ}F$ ) on projects other than Projects B and C are assumed to have been exposed to a hot weather environment, that is, to air temperatures above the  $27^{\circ}C$  ( $80^{\circ}F$ ) maximum level permitted in ASTM Method C 31-83 for initial site curing. This group comprises 83% of all observations on concretes of more than  $32^{\circ}C$  ( $90^{\circ}F$ ) placing temperature.

On all the sites, those specimens molded on Fridays or on days before holidays were left at the site until the next working day. Identification of these specimens depended on indications of the "date received in the laboratory" on test reports. This information was not shown on all the reports. From data series with complete information, it is inferred that the site curing period exceeded 24 h for about 15% of the specimen sets. The standard requirement of providing specimens with an impervious cover during initial site curing was generally adhered to.

Laboratory data on plain concrete that permitted a separate evaluation of the effect of high initial curing temperatures compared with the effect of high mixing temperatures indicate that the curing temperatures have a more detrimental effect on strength [2, 4-6]. The performance of concrete specimens on projects with nonstandard site curing, therefore, needs to be judged in terms

of adverse early curing conditions implied by the higher placing temperature levels recorded on the projects.

# **Statistical Evaluation**

## Temperature-Strength Correlation

The correlation analysis of the data indicated no significant relationship between the placing temperatures and 28-day strengths on five of the projects, as evidenced by correlation coefficients (r) near zero (Table 3). In these instances, the temperature was thus shown to have had no noticeable influence on the compressive strength; that is, higher placing temperatures did not result in significantly lower concrete strengths. If "critical values of r" [7] are taken into account, a significant correlation is found to exist for two of the data series—in Project A at the 99% confidence level at 95 degrees of freedom (df) and in Project F at the 95% confidence level at 102 df. The regression equations for the two data series indicate that strength increases with increasing temperatures, at the rate of 1.7 MPa (244 psi) on Project A and 1.8 MPa (258 psi) on Project F, with each rise in placing temperatures of 5.6 degrees Celsius (10 degrees Fahrenheit).

Although statistical theory considers the fitting of regression lines inappropriate for variables with no significant relationship, regression lines were nevertheless inscribed in the remaining scatter diagrams to determine what, if any, bias could be discerned. The presence of curvilinear relationships was considered in these instances, but it did not improve the goodness of fit. Three of the regression lines indicate a slight increase in strength with increasing temperatures; one reveals no change; and that for the 48-MPa (7000psi) concrete on Project G shows a very moderate decline in strength, equal to

Project	Correlation Coefficient	Significance	Effect of Higher Placing Temperatures on Strength
A	0.28	significant correlation at the 99% confidence level at 95 df	strength increase
В	0.07	no significance	no effect
С	0.01	no significance	no effect
D	0.05	no significance	no effect
Е	0.12	no significance	no effect
F	0.21	significant correlation at the 95% confidence level at 102 df	strength increase
G	0.08	no significance	no effect

	[ABLE 3—Statistica	l evaluation of	f temperature-strength	correlations
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strength reductions of 0.3 MPa (43 psi) for each increment of 5.6 degrees Celsius (10 degrees Fahrenheit) in placing temperature.

In summary, correlation analysis of the temperature and strength variables showed no correlation for most of the series. When a moderate degree of significance was attained, the analysis revealed an increase in strength with increasing placing temperatures.

## Effect of Placing Temperatures Above 32°C (90°F)

The strength performance of concrete was examined for statistically significant differences in average strength and standard deviation between two groups of data within each series: those relating to concrete temperatures of  $32^{\circ}C$  (90°F) or less and data obtained at concrete placing temperatures above  $32^{\circ}C$  (90°F). The Project C data are not included because of a maximum temperature limit on concrete of  $32^{\circ}C$  (90°F). Using applicable equations for differences between means [8], no statistically significant differences were found between the strength averages of the two groups (Table 4), except on Project F. In this case, the average strength was significantly higher at the higher temperature level of the concrete.

In recognition of a possible greater data scatter at higher temperature levels of concrete to be expected from a greater frequency of site water additions, the strength uniformity at the two temperature levels was analyzed for significant differences in standard deviations. Using the F test for differences between variances (that is, the squares of standard deviations), no significant differences were found to exist between the standard deviations of the two temperature ranges of concrete, except in Project D. In this instance, there was a significant improvement in strength uniformity, at the 0.05 significance level, in concrete with placing temperatures above  $32^{\circ}C$  (90°F).

In summary, placing temperatures above 32°C (90°F) caused no significant changes in the average strength and standard deviation from those obtained in concrete at the lower temperature level, with two exceptions, which indicated an improved performance at higher placing temperatures.

## **Discussion of Evaluation Results**

The evaluation was confined to the effects of hot weather conditions on strength, disregarding possible impairment of concrete performance in other respects. Information and data available from the projects failed to indicate any problems, such as handling and placing difficulties or postplacement defects. The absence of changes in the concrete composition or requirements on placing temperature indicate that there were no major problems. A separate study, now in progress, deals with the effect on strength, under hot weather conditions, of additions of water at the job site, length of delivery time, and extended site curing for a limited data series. Of these variables, only delivery
TABLE 4–Statistical Evaluation of differences between average strengths and standard deviations at concrete temperatures of 90°F and below. and concrete temperatures above  $90^{\circ}F$ .<sup>a</sup>

	Test	(A) on Concrete of 90°F Lower Temperature	and	Ţ	(B) Tests on Concrete of smperature Above 90	<b>Ľ.</b> °	Significant Between (. (95% confi	Differences A) and (B) dence level)
Project <sup>b</sup>	Number of Tests	Average 28-Day Strength, psi	Standard Deviation, psi	Number of Tests	Average 28-Day Strength, psi	Standard Deviation, psi	Average Strengths	Standard Deviations
Α	57	5637	438	40	5759	435	ou	ou
В	192	5444	349	46	5457	348	ou	оп
D	165	4891	589	81	4855	489	ou	yes
ш	37	4851	372	21	5000	327	ou	ou
ц	16	5738	426	13	6102	47S	yes	no
IJ	220	8188	475	73	8153	485	ou	no
<sup>a</sup> Degree C( <sup>b</sup> Project C	elsius = (degre is not included	se Fahrenheit – 32)/ 1. The concrete was s	(1.8; 1000  psi = 0.000  psi	6.895 MPa. num limit on p	lacing temperature o	f 90°F.		

times in excess of 60 min and extended site curing were found to result in a moderate loss in average strength.

The data evaluation presented in this paper confirms the findings of earlier random observations of minimal adverse effects of hot weather conditions on the strength performance of set-retarded concrete. Adverse hot weather conditions included high placing temperatures and, for a portion of specimen sets, exposure to high temperature during the initial site curing. A common characteristic of the concrete mixtures was inclusion of set retarders in all the concrete or in all the concrete at higher temperature levels.

The field data thus corroborate the findings of laboratory research regarding the favorable effect of retarding admixtures on concrete performance at elevated temperatures, including studies by Tuthill and Cordon [9], Tuthill, Adams, and Hemme [10], Berge [11], and, most recently, Gaynor, Meininger, and Khan [5]. In some respects, the data by Gaynor et al may not fully reflect the effectiveness of set retarders in hot weather concrete because of a lower admixture dosage than is normally used in field concrete.

Little information is offered in pertinent literature on the mechanics by which set-retarding admixtures function to minimize the adverse effect on strength of elevated temperatures. In investigating the development of structure and strength in portland cement paste, Richartz [12] observed that retardation of the setting process of pastes by either lower temperatures or chemical admixtures produced the same system of long-fibered calcium silicate hydrates in early hydration phases in the available gel pore space, as is shown in Fig. 8 [13]. These fibers spanned the pore space and provided a structure that was subsequently interconnected by additional hydration products. On the other hand, set acceleration by either high temperatures or set-accelerating admixtures caused the rapid formation of short-fibered calcium silicate hydrates, as illustrated in Fig. 9 [14]. These short fibers initially failed to span the available pore space, although with sufficient cementitious material, this was accomplished at a later stage. At the same time, they restricted the development of longer fibers. The photo micrographs were made on tricalcium silicate pastes to provide better detail of the crystalline structures involved. Richartz concluded that set retardation, by whatever means it is achieved, causes formation of a higher proportion of interlocked long fibers in the paste structure and that, with the same degree of hydration and the same paste porosity, this type of structure contributes to a higher ultimate strength than structures consisting of a high proportion of short fibers.

### Conclusions

1. Evaluation of a large number of field test reports indicated that concrete which included a set-retarding admixture had strength performance characteristics at elevated placing and site curing temperatures that did not change from those of concrete used at lower temperatures.



FIG. 8—Transmission electron microscopic micrograph of a hardened tricalcium silicate paste with a set retarder at seven days age. The setting was retarded by the addition of 1% zinc oxide (water/cement ratio = 0.44 [13]).

2. The capability of the concrete to remain unaffected in its strength performance by the potentially adverse hot weather conditions is attributed to the inclusion of a set-retarding admixture in all concrete under hot weather conditions. The results of this evaluation corroborate the beneficial effect ascribed to set-retarder use in the American Concrete Institute report on hot weather concreting (ACI 305R-77) [15]. They also agree with pertinent findings of laboratory investigations into the properties of set-retarded concrete mixed and initially cured at elevated temperatures.



FIG. 9—Transmission electron microscopic micrograph of a hardened tricalcium silicate paste with set accelerator at seven days age. The setting was accelerated by the addition of 1% calcium chloride (water/cement ratio = 0.44 [14]).

3. Microscopic studies of hardened cement-water paste indicated that set retardation by chemical admixtures produces microstructures beneficial to strength which are similar to those produced by a lowering of paste temperature. A similar effect is implied when set retarders are used in concrete.

4. The data on plain concrete mixed at low temperatures and then initially cured at elevated temperatures show a significant strength reduction in relation to concrete maintained at its low mixing temperature during the initial curing period [2, 5, 6]. On the other hand, this data evaluation indicates that

set-retarded concrete placed and also initially cured at elevated temperatures is immune to adverse high-temperature effects on strength. Under hot weather conditions, it appears that a greater benefit to the strength performance of concrete accrues from the use of set retarders than from an artificial cooling of concrete.

5. When used with other suitable concrete components, set-retarding admixtures may, therefore, obviate the need for artificial cooling of most concrete under hot weather conditions, provided it has been demonstrated that the concrete of a given composition has performed satisfactorily in other respects besides strength.

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## Maturity Functions for Concrete Cured During Winter Conditions

**REFERENCE:** Naik, T. R., "Maturity Functions for Concrete Cured During Winter Conditions," *Temperature Effects on Concrete, ASTM STP 858*, T. R. Naik, Ed., American Society for Testing and Materials, Philadelphia, 1985, pp. 107-117.

**ABSTRACT:** The objective of this paper is to check the validity of the Nurse-Saul and the Arrhenius maturity functions for concrete cured under winter conditions.

The test specimens used for concrete compressive strength determination were 101.6 by 203.2-mm (4 by 8-in.) cylinders. (The measurements were made in English units.) These specimens were cured at nominal temperatures of 2.8, 12.8, and 22.8°C (37, 55, and 73°F). The cylinders were cast from concrete with water-to-cement ratios of 0.50, 0.60, and 0.70. Tests for compressive strength were performed at the ages of 12, 18, 24, 36, 48, 72, 120 (five days), and 168 h (seven days). The maturity and temperature of the cylinders were recorded at each test age. Curves were plotted for the compressive strength versus the relative maturity at 20°C for each water-to-cement ratio and for different curing temperatures. It was determined that the Arrhenius function gives better correlation between the relative maturity and the strength than the Nurse-Saul function. The author concludes that the Nurse-Saul function should not be used for maturity-strength determination under winter curing conditions.

**KEY WORDS:** concrete, temperature effects, maturity function, winter curing, compressive strength, *in situ* strength

Time and temperature are two of the most important factors that influence the strength of a given batch of concrete. The combined effect of time and temperature has been studied since 1904. It was not until the early 1950s, however, that McIntosh [1], Nurse [2], Saul [3], Bergstrom [4], and others [5] published reports relating time and temperature to the compressive strength of concrete. The time-temperature relationship is called maturity, which is defined as a function of time and temperature. Many maturity functions have been proposed since the early 1950s. The purpose of this paper is to check the

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validity of the Nurse-Saul maturity function [3], which is the most widely used, and compare it with the Arrhenius maturity function [6].

### **Background Information**

### Maturity Functions

Many different maturity functions relating temperature to time have been proposed since the early 1950s. A compilation of most of the functions proposed prior to 1970 was made by Malhotra in 1971 [7]. Byfors [8] has examined four functions that show the variation with the curing temperature. The two functions examined in this paper are the Nurse-Saul (Eq 1) and the Arrhenius (Eq 2) maturity functions, f(T)

$$f(T) = k_1 (T_c + 10) \tag{1}$$

$$f(T) = k_2 \exp\left(-\frac{E}{RT_k}\right)$$
(2)

where

 $k_1$  and  $k_2$  = proportionality constants,

 $T_c$  = temperature of concrete in degrees Celsius,

 $T_k$  = temperature of concrete in degrees Kelvin,

- E = activation energy in kilo joules per mole, and
- R = universal gas constant.

The Nurse-Saul function shown in Eq 1 uses  $-10^{\circ}$ C as the datum temperature below which it is assumed that there is no increase in the concrete strength with time. At this temperature, therefore, the increase in the maturity is zero. The Nurse-Saul equation has been well accepted for determining the maturity of concrete [7,9]. In 1953 Bergstrom [4] analyzed the published data [1-3,5,10-12] and concluded that:

It is evident that the parameter suggested by Nurse and Saul is well suited for determining the strength of a given concrete mix at varying ages and temperatures.

Recent tests performed by Byfors [8] in Sweden indicate that the Nurse-Saul function gives very poor representation of the maturity of concrete at low curing temperatures. Freiesleben-Hansen and Pedersen [6] have proposed Eq 2, which is based on the Arrhenius function for thermal activation. This function has proved capable of taking into account the influence of temperature within a wide range: -10 to  $+80^{\circ}$ C.

The hydration of cement in concrete is an exothermic reaction. The Arrhe-

nius equation takes into consideration the activation energy for the hydration process. Since the reaction is exothermic, for each temperature there is a slightly different activation energy, E. Also, different cements have different compositions, and hence the activation energy depends on the type of cement used. Freiesleben-Hansen and Pedersen [6] found that the activation energy concept could be used to predict values of E by using the following expressions

at 
$$T_c \ge 20^{\circ}$$
C,  $E(T_c) = 33.3$  kJ/mol  
at  $T_c \le 20^{\circ}$ C,  $E(T_c) = 33.3 + 1.47(20 - T_c)$  kJ/mol

Thus, the function, f(T), in Eq 2 can be evaluated for different temperatures.

### **Relative Maturity**

In order to obtain a comparison of the two maturity functions, it is necessary that the two functions be compared at the same datum temperature, usually 20°C. Let f(T) equal the maturity value at any time, t, where T is the constant temperature. Then, according to the Nurse-Saul function, if the temperature of the concrete specimens is constant, then

$$f(T) \cdot t = f(20^{\circ}\text{C}) \cdot t_{20}$$
$$t_{20} = \frac{f(T) \cdot t}{f(20^{\circ}\text{C})}$$

But

$$f(T) = k(T_c + 10) \qquad (\text{from Eq 1})$$

Thus, at  $T = 20^{\circ}$ C,  $f(20^{\circ}$ C) = 30 k, in degrees Celsius minus hours. Therefore

$$t_{20} = \frac{T_c + 10}{30} \cdot t$$

or

$$t_{20} = \int_{0}^{t} \frac{T_{c} + 10}{30} \cdot dt \tag{3}$$

For various values of  $T_c$  and t,  $t_{20}$  is calculated and can then be plotted against the corresponding concrete compressive strength. Of course,  $t_{20}$  is the

time needed to reach an equivalent maturity at 20°C. It also designates the relative maturity at 20°C in hours.

According to the Arrhenius function, if the curing temperature is constant, then

$$t_{20} = \frac{f(T) \cdot t}{f(20)}$$

But

$$f(T) = k \exp\left(-\frac{E}{RT_k}\right)$$
 (from Eq 2)

At  $T = 20^{\circ}$ C,  $f(20^{\circ}) = k \exp(-E/293R)$ . Therefore

$$t_{20} = \left[\frac{k \exp\left(\frac{-E}{RT_k}\right)}{k \exp\left(\frac{-E}{293R}\right)}\right] \cdot t$$

Thus

$$t_{20} = \exp\left[\left(\frac{E}{R}\right)\left(\frac{1}{293} - \frac{1}{T_k}\right)\right] \cdot t$$

or

$$t_{20} = \int_0^t \exp\left(\frac{E}{R}\right) \left(\frac{1}{293} - \frac{1}{T_k}\right) \cdot dt \tag{4}$$

Equation 3 gives the value of relative maturity, in hours, by the Nurse-Saul function, and Eq 4 gives the value of relative maturity, in hours, by the Arrhenius function.

## **Materials and Methods**

The test specimens used for concrete compressive strength determination were 101.6 by 203.2-mm (4 by 8-in.) cylinders. (The measurements were made in English units.) These specimens were cured at nominal temperatures of 2.8, 12.8, and 22.8°C (37, 55, and 73°F). The cylinders were cast from concrete with water-to-cement ratios of 0.50, 0.60, and 0.70. Tests for compressive strength were performed at the ages of 12, 18, 24, 36, 48, 72, 120 (five

days), and 168 h (seven days). The maturity and temperature of the cylinders were recorded at each test age. Curves were plotted for the compressive strength versus the relative maturity at 20°C for each water-to-cement ratio and for different curing temperatures.

## Results

For each mix, the compressive strength and the temperature of the concrete cylinders were recorded at 12, 18, 24, 36, 48, 72, 120, and 168 h. The  $t_{20}$ value was calculated at each of these test ages and was plotted against the compressive strength, as shown in Figs. 1 through 6.



FIG. 1—Compressive strength versus relative maturity of concrete, using the Nurse-Saul function at water/cement ratio (W/C) = 0.50. See text for details.



FIG. 2—Compressive strength versus relative maturity of concrete, using the Nurse-Saul function at W/C = 0.60. See text for details.

### **Discussion of Results**

Figures 1 through 6 present the results of the concrete compressive strength versus the relative maturity, at  $t_{20}$ , for specimens cured at 2.8, 12.8, and 22.8°C (37, 55, and 73°F). In Figs. 1, 2, and 3 the relative maturity is based on the Nurse-Saul function. An examination of these three figures indicates that at the curing temperatures of 12.8 and 22.8°C (55 and 73°F), the Nurse-Saul function has very good correlation with the compressive strength. At the lowest curing temperature of 2.8°C (37°F), however, it has a very poor correlation with compressive strength. This indicates that the Nurse-Saul function does not fully represent the variation of temperature with compressive strength, particularly at low curing temperatures.



FIG. 3—Compressive strength versus relative maturity of concrete, using the Nurse-Saul function at W/C = 0.70. See text for details.

In Figs. 4, 5, and 6 the relative maturity is based on the Arrhenius function. These figures clearly show that at all curing temperatures [2.8, 12.8, and 22.8°C (37, 55, and 73°F)] there is a very good correlation between compressive strength and relative maturity. The plots of the Arrhenius function show that for a given mix with a certain water-to-cement ratio (W/C) and cured at any temperature from 2.8 to 22.8°C (37 to 73°F), the same strength is reached at the same maturity, irrespective of its curing temperature history.

## Conclusion

The results of this paper indicate that the Arrhenius function gives a much better maturity-strength relationship than the Nurse-Saul function over a



FIG. 4—Compressive strength versus relative maturity of concrete, using the Arrhenius function at W/C = 0.50. See text for details.

wide range of concrete curing temperature variations. The Nurse-Saul function can be used for relatively higher curing temperatures, but it certainly should not be used for winter curing conditions.

## Recommendations

The Arrhenius function is recommended for use when the concrete is subjected to winter curing, as well as to other curing temperatures. The Nurse-Saul function should be used only for relatively high curing temperatures, since the predicted strength for these temperatures is about the same when either of these two maturity functions is used.



FIG. 5—Compressive strength versus relative maturity of concrete, using the Arrhenius function at W/C = 0.60. See text for details.



FIG. 6—Compressive strength versus relative maturity of concrete, using the Arrhenius function at W/C = 0.70. See text for details.

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# Temperature Effects on Strength and Elasticity of Concrete Containing Admixtures

**REFERENCE:** Nasser, K. W. and Chakraborty, M., "Temperature Effects on Strength and Elasticity of Concrete Containing Admixtures," *Temperature Effects on Concrete*, *ASTM STP 858*, T. R. Naik, Ed., American Society for Testing and Materials, Philadelphia, 1985, pp. 118-123.

**ABSTRACT:** This paper investigates the influence of temperature on the structural properties of sealed and unsealed concrete containing Saskatchewan lignite fly ash and sulfonated naphthalene formaldehyde superplasticizer. Concrete was exposed for periods of up to six months to temperatures of -11 to  $232^{\circ}$ C (12 to  $450^{\circ}$ F).

Results show that temperatures up to  $71^{\circ}$ C (160°F) have a minor effect on both the strength and elasticity of sealed or mass concrete; however, exposed unsealed concrete showed a gradual increase in strength and minor changes in elasticity under the same conditions. At a temperature increase of 121 to 232°C (250 to 450°F), the strength and elasticity of mass concrete decreased by about 65%, while the strength of exposed unsealed concrete was not affected; however, its elasticity was reduced by about 40% only.

Naphthalene superplasticizer admixtures do not seem to influence the properties of hardened concrete containing fly ash and exposed to high temperatures.

**KEY WORDS:** admixtures, concrete, elasticity, fly ash, hardened concrete, mass concrete, sealed concrete, concrete strength, unsealed concrete, temperature

Admixtures, such as fly ash and high-range water reducer Type F [ASTM Specification for Chemical Admixtures for Concrete (C 494-82)], commonly known as superplasticizers, are used in concrete structures in many projects. Fly ash has recognized helpful effects, such as low heat of hydration, improved workability, and resistance to sulfates and other destructive agents. But the presence of fly ash in concrete retards the rate of strength development and may increase drying shrinkage and creep. Superplasticizers are used in fresh concrete, either to reduce the water-to-cement ratio in the mix while maintain-

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ing the same workability or to increase the workability of a mix without changing its water-to-cement ratio. Therefore, use of superplasticizers in concrete containing fly ash is being considered favorably in the construction industry. So far there have been no reports of adverse effects of superplasticizers in hardened concrete at normal temperatures. However, very little is known about their effects on strength and elasticity at low or high temperatures. Considerable research has been done [1-5] on concrete, both sealed and unsealed, with or without fly ash, at elevated temperatures. Consequently, there is great interest in the effects of temperature on the properties of superplasticized concrete containing fly ash, since such a concrete mix can be used to advantage in pressure vessels of nuclear reactors and in other structures exposed to a wide range of temperatures.

The present investigation was carried out to study the effect of temperature on sealed and unsealed air-entrained concrete containing fly ash, conventional water reducer, and superplasticizer. The properties of strength and elasticity were studied at seven different temperatures ranging from -11 to  $232^{\circ}$ C (12 to  $450^{\circ}$ C) and at seven different exposures of 1 to 180 days. The results are presented in the following pages. Sealed concrete will here after be referred to as mass concrete.

### **Test Program**

The tests were made on easy-to-handle 7.6 by 23.5-cm (3 by 9<sup>1</sup>/<sub>4</sub>-in.) concrete cylinders using sulfate-resisting portland cement (ASTM Type V). A 25% replacement of cement by Saskatchewan lignite fly ash was used on the basis of weight. The results of chemical and physical analysis of fly ash are given in Table 1. Saskatchewan lignite fly ash conforms to some of the specifications of the ASTM Specification for Fly Ash and Raw or Calcined Natural Pozzolan for use as a Mineral Admixture in Portland Cement Concrete (C 618-83) for Type C fly ash. Local crushed aggregates of 1.9-cm (<sup>3</sup>/<sub>4</sub>-in.) maximum size, composed mostly of a mixture of dolomite and hornblende, were used. A ratio of aggregate to cement plus fly ash, A/(C + FA), of 7.5 and a ratio of coarse to fine aggregate of 1.15 were used in the concrete mix, as outlined in Table 2. A vinsol resin air-entraining agent of 1.11 mL/kg (0.5 mL/lb) of cement and a conventional water reducer of 2.9 mL/kg (1.3 mL/lb) of cement were used in the mix. The ratio of water to cement plus fly ash, W/(C + FA), was kept at 0.6, which was also used in earlier investigations [3,4]. A superplasticizer of sulfonated naphthalene formaldehyde condensates which has a dark brown color and specific gravity of 1.2 was used in the mix. This superplasticizer satisfies the requirements of ASTM Standard C 494-82 for high-range water reducer Type F.

The concrete was mixed in a laboratory pan mixer while the superplasticizer was added in accordance with the ASTM Standard for Making and Curing Concrete Test Specimens in the Laboratory (C 192-81). Air content and

Property	Average of 3 Samples
Chemical Composition, %	
Silica (SiO <sub>2</sub> )	43.03
Mixed oxides $(R_2O_3)$	27.93
Magnesium (MgO)	3.67
Sulfur (SO <sub>3</sub> )	0.59
Ignition loss	0.55
Available alkali (NO <sub>2</sub> O)	2.56
Physical properties	
Specific gravity	2.30
Blaine fineness, $cm^2/g$	22.53
Autoclave soundness expansion, %	0.061
Compressive strength of mortar cubes	
% of control at 7 days	133
% of control at 28 days	145
Drying shrinkage of mortar bars at 28 days, %	0.089

 
 TABLE 1—Chemical composition and physical properties of Saskatchewan fly ash.

TABLE 2-Mix Proportions of concrete containing fly ash.<sup>a</sup>

	Mix Proportions								
– Composition	lb	g	lb/yd <sup>3</sup>	kg/m <sup>3</sup>					
Cement	11	4 989	338.8	196.3					
Fly ash	2.75	1 247	84.7	49.1					
Cement and fly ash	13.75	6 236	423.5	245.4					
Water	8.18	3 710	251.9	146.0					
Fine aggregate	47.25	21 430	1 455.2	843.2					
Coarse aggregate	55.75	25 430	1 716.9	1 000.5					
Sulfonated naphthalene									
superplasticizer	0.165	75	5.08	2.95					
Pozzolith		16	1.08	0.63					
Vinsol air entrainment agent		12	0.81	0.47					

<sup>a</sup>Ratios:

W/(C + FA) = 0.6A/(C + FA) = 7.49

FA/CA = 1.15

K-slump values of the fresh concrete were determined before the superplasticizer, in the proportion of 1.2% by weight of the cement, was added to the fresh concrete. The concrete was further mixed for 2 min, and new air-content and slump tests were performed before the specimens were cast.

Twelve specimens at a time were cast in cast iron molds. The specimens were compacted in three layers by rodding each layer ten times. This procedure was chosen after some trial and error tests were performed for best results. It was observed that noncompacted specimens contained a considerable number of honeycombs of varying sizes whereas standard compaction led to excessive segregation, resulting in local failure at the top of the specimen during testing. The day after casting, the specimens were demolded, weighed, and marked before they were placed in water to be standard cured for 28 days. The mass concrete specimens that were cured at 21°C (70°F) or less were cast in plastic jackets sealed by glueing aluminum lids at each end of the cylinder. The specimens exposed to temperatures higher than 21°C (70°F) were sealed in hollow steel cylinders by welding [2] so that they might withstand the high temperature without significant loss of moisture. The mass concrete specimens at 21°C  $(70^{\circ}F)$  were kept in a water bath while those at  $-11^{\circ}C$  (12°F) were kept in an antifreeze bath. All the specimens at other temperatures were kept in electric ovens. The specimens were exposed to seven different temperatures of -11, 21, 71, 121, 149, 177, and 232°C (12, 70 160, 250, 300, 350, and 450°F). At each temperature, a minimum of three specimens were tested for strength and elasticity. Standard-cured specimens were exposed to different temperatures for periods of 1, 3, 7, 14, 28, 90, and 180 days. All the specimens were brought to room temperature before testing. The strength and elasticity tests were conducted according to the ASTM Test for Compressive Strength of Cylindrical Concrete Specimens (C 39-83a) and ASTM Test for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression (C 469-81).

### **Test Observations and Results**

The average slump of the concrete mixes before the addition of superplasticizer was 5.2 cm (2 in.) and it increased to 19.05 cm (7.5 in.) thereafter. The air content was observed to be 6.25% before and 4.8% after the addition of superplasticizer. The average 28-day compressive strength and elasticity for the mix at 21°C (70°F) were 24.82 MPa (3600 psi) and 24.5 GPa (3.55 × 10<sup>6</sup> psi), respectively. The ratio of compressive strength,  $f_c$ , at different temperatures and exposures to that at 21°C (70°F) and 28 days,  $f'_c$ , are given in Tables 3 and 4 for mass and unsealed concrete, respectively. Similarly, the modulus of elasticity ratios,  $E/E_c$ , are given in Tables 5 and 6 for mass and unsealed concrete. Figures 1 and 2 show the variation of strength ratios for mass concrete,  $f_c/f'_c$ , against temperature and different exposures while Figs. 3 and 4 show the elasticity ratios,  $E/E_c$ . The variation of strength and elasticity ratios against temperature for unsealed concrete is shown in Fig. 5, and the same variation for different exposures and temperatures is given in Fig. 6.

### Loss of Moisture and General Remarks

A typical specimen of the present concrete mix contained about 185 mL of water at the time of casting. The loss of moisture content in the case of mass

	$f_c/f_c$ at Indicated Temperature						
Exposure Time, days		21°C (70°F)	71°C (160°F)	121°C (250°F)	149°C (300°F)	177°C (350°F)	232°C (450°F)
1	1.02	1.00	1.01	0.95	0.98	0.95	0.72
3	1.00	1.05	1.06	1.05	0.83	0.68	0.57
7	1.07	1.11	1.10	1.15	0.92	0.80	0.66
14	1.07	1.16	1.22	1.17	0.83	0.76	0.60
28	1.09	1.25	1.07	1.21	1.15	0.76	0.55
90	1.00	1.33	1.07	1.44	1.25	0.54	0.41
180	1.05	1.42	1.04	1.22	0.80	0.44	0.34

 TABLE 3—Ratio of compressive strengths, f/f<sub>c</sub>, of mass concrete at various temperatures and exposures and at 21°C (70°F) and 28 days, 24.82 MPa (3600 psi).

TABLE 4—Ratio of compressive strengths.  $f_c/f_c$ , of exposed unsealed concrete at various temperatures and exposure and at 21 °C (70 °F) and 28 days, 24.82 MPa (3600 psi).

		$f_c/f_c'$ at Indicated Temperature						
Exposure Time, days		21°C (70°F)	71°C (160°F)	121°C (250°F)	149°C (300°F)	177°C (350°F)	232°C (450°F)	
1	1.00	1.05	1.17	1.22	1.27	1.25	1.20	
3	1.04	1.19	1.15	1.35	1.31	1.26	1.26	
7	1.06	1.27	1.21	1.23	1.23	1.17	1.23	
14	1.08	1.28	1.34	1.32	1.20	1.23	1.17	
28	1.00	1.28	1.28	1.21	1.25	1.27	1.25	
90	1.09	1.27	1.23	1.16	1.13	1.10	1.11	
180	1.15	1.18	1.22	1.11	1.08	1.05	0.98	

TABLE 5—Ratio of elasticity, E/E, for mass concrete at various temperatures and exposuresand at 21°C (70°F) and 28 days. 24.5 GPa (3.55 × 10<sup>6</sup> psi).

		$E_c/E_c$ at Indicated Temperature							
Exposure Time, days		21°C (70°F)	71°C (160°F)	121°C (250°F)	149°C (300°F)	177°C (350°F)	232°C (450°F)		
1	1.00	1.04	1.10	0.90	0.82	0.82	0.52		
3	1.00	1.04	1.02	0.77	0.86	0.58	0.45		
7	1.16	1.13	1.10	0.85	0.97	0.65	0.42		
14	1.16	1.18	1.14	1.03	0.84	0.62	0.41		
28	1.12	1.30	1.00	1.00	0.77	0.64	0.34		
90	1.01	1.41	1.15	0.81	0.72	0.43	0.32		
180	1.00	1.45	1.10	0.70	0.70	0.41	0.30		

		$E_c/E_c$ at Indicated Temperature							
Exposure Time, days	-11°C (12°F)	21°C (70°F)	71°C (160°F)	121°C (250°F)	149°C (300°F)	177°C (350°F)	232°C (450°F)		
1	1.03	1.00	1.00	0.97	0.87	0.79	0.62		
3	0.96	1.00	0.85	0.92	0.72	0.65	0.66		
7	1.04	0.98	0.92	0.83	0.75	0.68	0.67		
14	0.96	1.00	0.97	0.78	0.84	0.76	0.66		
28	0.88	1.00	0.80	0.70	0.84	0.77	0.70		
90	0.94	0.94	0.85	0.71	0.72	0.62	0.62		
180	0.81	0.94	0.93	0.86	0.80	0.65	0.57		

 TABLE 6—Ratio of elasticity, E/E<sub>c</sub>, for exposed unsealed concrete at various temperatures and exposures and at 21 °C (70°F) and 28 days, 24.5 GPa (3.55 × 10<sup>6</sup> psi).

concrete up to 71°C (160°F) for any duration of exposure was small. The loss of moisture at 121, 149, 177, and 232°C (250, 300, 350, and 450°F) and 90 days of exposure was observed to be 11.3, 15.1, 18.3, and 21.6% of the original water content, respectively. Similar values were observed in specimens after they were exposed for 1 day only at those temperatures. Moisture losses at 180 days were a little less. However, unsealed concrete exposed to high temperatures suffered higher losses of moisture starting 1 day after exposure. At temperatures of 71, 121, 149, 177, and 232°C (160, 250, 300, 350, and 450°F) the losses at 1 day were observed to be 36, 62, 67, 73, and 74%. The corresponding values at 180 days were 68, 71, 72, 76, and 78, respectively. The losses of moisture at temperatures of -11 and 21°C (12 and 70°F) and an exposure of 180 days were observed to be 15% and 49%, respectively.

Both mass and unsealed concrete at temperatures of  $121^{\circ}C$  ( $250^{\circ}F$ ) and higher turned whitish; the degree of whiteness increased with temperature, especially in mass concrete. Also, there was an odor of condensed steam in the mass concrete specimens. The odor became prevalent starting at temperatures of about  $121^{\circ}C$  ( $250^{\circ}F$ ). The stress-strain curve of specimens exposed to 21 to  $71^{\circ}C$  (70 and  $160^{\circ}F$ ) was typical of ordinary concrete. Specimens exposed to  $-11^{\circ}C$  ( $12^{\circ}F$ ) indicated lower ductility. All the specimens at temperatures beyond  $71^{\circ}C$  ( $160^{\circ}F$ ) showed greater strain with increased temperature. The failure of specimens under load was gradual rather than brittle. At 177 and  $232^{\circ}C$  (350 and  $450^{\circ}F$ ) the failure mode was slow and dull, and there was appreciable increase in strain prior to failure. This behavior was more pronounced for sealed specimens.

#### Compressive Strength and Elasticity of Mass Concrete

The compressive strength of mass concrete, as observed from Table 3 and Figs. 1 and 2, was practically unaffected by one-day heating except at  $232^{\circ}C$  (450°F), at which point the loss of strength was 28%. The strength of concrete





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FIG. 5-Relationship of strength and elasticity ratios and temperature for unsealed exposed concrete.





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at -11 and  $71^{\circ}$ C (12 and  $160^{\circ}$ F) did not increase significantly for any length of exposure. Beyond 14 days, the strength at  $121^{\circ}$ C ( $250^{\circ}$ F) was greater than the respective values at  $71^{\circ}$ C ( $160^{\circ}$ F). Between 121 and  $232^{\circ}$ C (250 and  $450^{\circ}$ F) the strength dropped monotonically.

Similar to compressive strength ratios, the modulus of elasticity ratios for mass concrete,  $E/E_c$ , at 21°C (70°F) were usually greater than the corresponding values at -11°C (12°F). Between 121°C (250°F) and 232°C (450°F), the temperature had a significant adverse effect on elasticity of mass concrete. The greatest reduction was always observed to be at 180 days. At 232°C (450°F) the elasticity ratio dropped continuously with the length of exposure, and this loss was more than the corresponding loss in strength. At 180 days the loss of elasticity at 177 and 232°C (350 and 450°F) was 59 and 70%, respectively, compared with its values at 28 days and 21°C (70°F). A higher temperature with a short exposure seemed to be more effective in reducing the modulus of elasticity than prolonged exposure at a lower temperature.

The observation that temperatures up to  $71^{\circ}C$  (160°F) have a minor effect on both the strength and elasticity of sealed specimens agrees with results of the previous investigations [3] of concrete made with portland cement with or without fly ash. In the present investigation, the maximum increase in strength was 44% at 121°C (250°F) in comparison with 52% under similar conditions for the earlier investigation [3].

## Compressive Strength and Elasticity of Unsealed Concrete

The strength of standard-cured, unsealed concrete exposed to high temperatures increased slightly. But the modulus of elasticity of the unsealed concrete, unlike the strength, was adversely affected by temperature, as shown in Figs. 5 and 6. Between 71 and  $232^{\circ}$ C (160 and  $450^{\circ}$ F), the modulus was observed to be less than the 28-day values at  $21^{\circ}$ C ( $70^{\circ}$ F). Although the reduction of the modulus with time up to 90 days did not follow any particular pattern, its trend was gradual because of both temperature and exposure.

## Discussion

Whenever concrete is subjected to high temperatures in a closed system, saturated steam pressure will surround the specimen. This condition will result in high pore pressure, which causes deterioration in the structural properties of the cement gel. Because most aggregates are inherently stable at a temperature of 232°C (450°F), the possibility of hydrothermal reactions between aggregates and cement is negligible.

The products of hydration of portland cement concrete under ordinary conditions are mainly silicates, aluminates, and lime. The hydrated lime, having no cementitious qualities of its own, is soluble in water but will react gradually with fly ash under normal conditions and form cementitious compounds. This secondary hydration results in added strength in the concrete at ordinary temperature.

Between 121°C (250°F) and 149°C (300°F), the mass concrete used in this investigation exhibited higher strength than the 28-day compressive strength at 21°C (70°F). This behavior is contrary to the results for mass concrete made with portland cement only [1, 2, 6]. The increase in strength is attributed to the secondary products of hydration between lime and fly ash. This product, which is a low-lime silicate, is different from tobermorite gel, a relatively highlime silicate which is produced at room temperature and atmospheric pressure [7]. The strength of tobermorite was reported to be two to three times higher than that for tobermorite gel. Also, it is known that for concrete containing fly ash, a very small amount of alpha-dicalcium silicate ( $\alpha \cdot 2CaO \cdot Si_2 \cdot H_2O$ ) is formed at temperatures of about 100°C (212°F) [1,2]. This compound is highly crystalline, porous, and a very poor binder. At higher temperatures, presumably a greater portion of tobermorite and tobermorite gel can be transformed into alpha-dicalcium silicate, resulting in greater deterioration in the structural properties of the concrete. Therefore, the increase in strength of mass concrete up to certain temperatures and exposures and the decrease in strength with higher temperatures can be attributed to the effect of these secondary hydration products.

The continuous increase in the modulus of elasticity with time at  $21^{\circ}$ C (70°F) as shown in Fig. 4, indicates that the tobermorite gel is likely to have elastic properties similar to those of the cement gel products. At 121 and 149°C (250 and 300°F) the increase in strength in Fig. 2 does not match the corresponding variation in elasticity in Fig. 4. This may suggest that, though tobermorite has higher strength, it does not have a corresponding higher elasticity. Also, the alpha-dicalcium silicates are known to have low strength and elasticity. All of this would explain why the lower values of elasticity ratios at higher temperatures did not match the corresponding values of strength ratios.

The strength of unsealed concrete was not affected at  $-11^{\circ}C$  (12°F) for any length of exposure. At temperatures from 21 to 232°C (70 to 450°F) there was a general trend for the strength to increase up to 28 days of exposure; at 21, 71, 121, 149, 177, and 232°C (70, 160, 250, 300, 350, and 450°F) the strengths were observed to increase by 28, 34, 32, 25, 27, and 25%, respectively. Thereafter, the strength gradually decreased up to 180 days, but the resultant strength at any temperature was always greater than the 28-day standard compression strength except at 232°C (450°F) at which point it was lower by 2% only. The extent of increase in strength of exposed concrete appears to be slightly greater than the earlier observed values [4] for ordinary portland cement concrete with fly ash only.

The elasticity of unsealed concrete at -11 and  $21^{\circ}$ C (12 and  $70^{\circ}$ F) seemed to be unaffected by the length of exposure at exposures up to six months. The elasticity in general started to decrease with temperature at temperatures from 71 to 232°C (160 to 450°F) and with exposure up to 180 days. The elasticity values at 71, 121, 149, 177, and 232°C (160, 250, 300, 350, and 450°F) were observed to be 80, 70, 72, 62, and 57%, respectively.

The small increase in the strength of unsealed concrete could be attributed to drying and to some secondary hydration applicable within the limits of temperature and exposure. On the other hand, the reduction of the modulus of elasticity with temperature might be due to both induced cracking with drying and transformation of the gel products into alpha-dicalcium silicates.

## Conclusions

The following preliminary conclusions can be drawn from the present investigation concerning the effect of temperature on mass and exposed air-entrained concrete containing admixtures of fly ash, water reducer, and superplasticizer:

1. For any length of exposure up to six months, the strength and elasticity of mass concrete at -11 and  $71^{\circ}$ C (12 and  $160^{\circ}$ F) were almost equal to their corresponding values at  $21^{\circ}$ C ( $70^{\circ}$ F). This result is in agreement with previous investigations for concrete made with portland cement with or without fly ash.

2. Water-cured concrete specimens at  $21^{\circ}$ C (70°F) continued to gain strength and elasticity with age. After six months of exposure the strength and elasticity values increased by 42 and 45%, respectively, in comparison with their respective values at 28 days.

3. At  $121^{\circ}C$  ( $250^{\circ}F$ ) the compressive strength of mass concrete was greater than its 28-day strength, and in one case it was 44% higher. However, the modulus of elasticity was generally less than that at 28 days. This behavior is different from that of portland cement concrete, and it may be attributed to the formation of tobermorite in the presence of fly ash in concrete.

4. At  $149^{\circ}C(300^{\circ}F)$  the strength of mass concrete after 28 and 90 days of exposure was greater than the 28-day strength at  $21^{\circ}C(70^{\circ}F)$ ; thereafter, the strength decreased. The elasticity, on the other hand, was always lower than the corresponding value at 28 days.

5. Immediately after one day of exposure to temperatures of 177 and  $232^{\circ}C$  (350 and 450°F), both the strength and elasticity of mass concrete were reduced. The strength, after an exposure of six months, was reduced to 44 and 34%, respectively, while the elasticity was reduced to 41 and 30% of its value at 28 days and 21°C (70°F). These values are in reasonable agreement with earlier results obtained from concrete containing normal portland cement and fly ash exposed to the same conditions. The deterioration of the structural properties at high temperature is attributed to the transformation of most of the tobermorite and tobermorite gel into crystalline alpha-dicalcium silicates which have lower strength and elasticity.

6. The strength of unsealed concrete at  $-11^{\circ}C(12^{\circ}F)$  was not significantly changed by the length of exposure. At temperatures from 21 to 232°C (70 to 450°F) the strength of unsealed concrete increased in general up to 28 days; thereafter, it decreased slightly but did not go below the 28-day strength at 21°C (70°F).

7. The elasticity of exposed concrete at -11 and  $21^{\circ}$ C (12 and  $70^{\circ}$ F) was not altered significantly for any exposure period. At temperatures from 71 to 232°C (160 to 450°F) the value of elasticity decreased in general with both temperature and exposure. The elasticity at 177 and 232°C (350 and 450°F) and 180 days was reduced to 65 and 57% of its original value at 28 days and 21°C (70°F).

8. Naphthalene superplasticizer admixtures do not seem to change the properties of hardened portland cement concrete containing fly ash and exposed to high temperatures.

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# Effect of Temperature Rise and Fall on the Strength and Permeability of Concrete Made With and Without Fly Ash

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**ABSTRACT:** The effect of temperature rise and fall on concrete at early ages is rarely measured. Concrete cured at elevated temperature has reduced strength at later ages. The changes that occur in the hydration products have been measured in terms of the amount of calcium hydroxide  $[Ca(OH)_2]$  present. Since permeability is, to some degree, a function of the water/cement ratio and strength, the measurement of permeability was necessary. Concrete is more permeable after it has undergone a temperature cycle similar to that found in practice. The use of Class F fly ash can effectively reduce the permeability of heat-affected concrete.

**KEY WORDS:** concrete, portland cement, fly ash, standard tests, chemical analysis, heat of hydration, exothermic reaction, temperature cycle, water/cement ratio, strength, carbonation, alkali-aggregate reaction, cement technology, Bogue compounds, calcium hydroxide  $[Ca(OH)_2]$ , concrete permeability, permeability tests

The development of standard test methods performed under controlled conditions, particularly for assessing the strength performance of different sources of portland cement or for determining the "in source" variability, has been a preoccupation not only of cement producers but also of users. Much effort has been directed toward the standardization of methods of testing, unrelated to conditions that occur in practice. When concrete is defective, the cause is usually too high a water/cement ratio (W/C) or damage from alkali-aggregate reaction (AAR).

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Carbonation can be a problem that is difficult to recognize for, although concrete with a high W/C may have adequate structural strength, a superficial inspection of the structure gives no indication of the amount of protection provided to the reinforcement. The influence that high strength or high heat has had on the later strength development of portland cement concrete, particularly after 28 days, has diverted attention away from the W/C and from why concrete is now generally made with a higher W/C than it was, say, 50 years ago [1]. Thus, consideration of permeability is important in the assessment of carbonation and durability.

It may seem unnecessary to be constantly reminded that the hydraulic reaction that takes place in concrete is exothermic and that the amount of heat produced usually causes the temperature of *in situ* concrete to rise considerably. However, the information is rarely used except for formwork removal. For instance, Harrison [2] has made temperature predictions for structural concrete placed in 18-mm plywood forms in a section width of just 300 mm. When the temperature of the concrete at the time of placing is 5°C and the mean ambient site temperature is 0°C, the *in situ* temperature can be expected to rise by 13 to 18 degrees Celsius. If the same element is cast when the placing temperature is 20°C and the mean ambient temperature 15°C, the concrete temperature can be expected to rise by 24 to 44 degrees Celsius. What would happen if, for example, the ambient temperature was 35°C was not calculated, but the concrete would probably experience a temperature rise exceeding 70 degrees Celsius. Portland cement concrete of the same mixture proportions, giving nominally the same strength under standard conditions, will have different performance characteristics at later ages because of the temperature regimens experienced at early ages.

It is the intention of this paper to show that this temperature rise and fall at early ages, caused by cement hydration, has some important side effects that reduce the performance of portland cement concrete and that the performance can be improved by the inclusion of fly ash. This temperature rise and fall will be referred to as the *in situ* temperature cycle (ITC).

Although the use of fly ash has important environmental implications [3], it is often used as part of the cementitious material content of concrete to reduce the size of the ITC. Research with *in situ* concrete has shown that, with particular grades of Class F fly ash, the reduction of temperature is not proportional to the amount of portland cement substituted and that, for the same concrete strength at 28 days with and without fly ash, the ITC will be practically the same, but the properties of the two types of concrete will be significantly dissimilar.

Bamforth [4] showed that for concrete in which 30% of a Type I portland cement was substituted by a Class F fly ash, of which the loss-on-ignition was 3.0% and the amount retained on a 45- $\mu$ m sieve was 5.6%, the reduction in temperature was less than 15%.

The concrete was proportioned for a 75-mm slump with a cementitious material content of  $400 \text{ kg/m}^3$ , with water/cementitious material ratios of 0.45 for

the portland cement and 0.41 for the fly ash concretes. The compressive strength at 28 days for standard and temperature-matched curing [5] (in which the test specimens are subjected to the same thermal history as the concrete in the element, that is, storing test specimens so that they follow the temperature history of a point at which a sensor is embedded in a structure) showed the following:

1. For standard curing, the strength of the fly ash concrete was 7% lower than that of the control concrete.

2. Compared with standard curing, the effect of the ITC on the portland cement concrete resulted in a reduction of 22%, while the fly ash concrete had increased 18%. More significantly, the strength of the fly ash concrete was 55% greater than that of the control concrete made with portland cement.

This research drew attention to some other effects of the ITC on the properties of concrete. Not only were the creep and shrinkage of the fly ash concrete reduced, but the portland cement concrete had a significantly lower Young's modulus. This showed that prestressed concrete would benefit significantly from the inclusion of fly ash. There are many references in the literature to the use and benefits of fly ash for high-strength concrete, but only limited research has been done on concrete subjected to an ITC or simulated conditions that approach those found in practice.

The justification for "high-heat" portland cements has not been sufficiently questioned for, although they reduce the formwork striking times and are required for use in precasting, the fact remains that concrete is principally made to a characteristic minimum 28-day strength.

The increase in the present performance of portland cement, in comparison with that of some 30 years ago in Britain [6] and 50 years ago in the United States [7], has been mainly due to modifications in the manufacture of portland cement and a general improvement in testing techniques for both cement and concrete. The changes in portland cement production have been generally brought about by alterations in the following:

- (a) preparation of the raw materials,
- (b) kilning technique,
- (c) rates of clinker cooling, and
- (d) milling.

The cumulative effect of the first three of these changes generally enables production of more alite—often incorrectly called tricalcium silicate ( $C_3S$ ) because that is what is calculated from chemical analysis—resulting in portland cements with typically 50 to 60%  $C_3S$ . In addition, modern air-swept, closed-circuit grinding techniques produce more uniform particle-size distribution of the milled cement. This does not significantly change the specific surface but causes the cement to hydrate more quickly and more thoroughly, thus producing greater early strength in concrete.

There are many cements available which, under standard curing condi-

tions, manifest rapid strength development. However, they do not appear to perform to the same advantage in *in situ* concrete, and the evidence suggests that, particularly where low water/cement ratios are specifically required for durability, they should not be used. In practice, if the concrete is kept moist, it is likely that such cements will produce a larger ITC, which makes it possible for most of the hydration to be completed within a few days, unlike standard moist curing conditions, in which significant hydration continues for months or even years.

A common misconception that has prevailed among most users has been that high-heat portland cements in concrete perform in a manner similar to that of cements of 50 years ago—that is, at 28 days the strength is about two thirds of that obtained at one year, provided moist conditions are maintained. In Britain the change has been recognized, much to the concern of the majority of users, in that the latest draft revision of the Code of Practice for reinforced concrete [8] does not make any recommendation for assuming increased strength after 28 days. This has led to the withdrawal of strength/aging factors for concrete that is made and stored in water at 20°C, that is, unless there is evidence to the contrary.

Because of the discovery of alkali-aggregate reaction (AAR) in concrete in the late 1930s in the United States [9] and in the 1970s in Britain [10], the relationship of the chemistry and physical nature of the products of hydration to the performance of concrete made with high-heat portland cements must be reconsidered. This is all the more important because either natural pozzolans or good-quality fly ash can be used to prevent the expansive reaction of AAR. The distinction between a good and a poor pozzolan is not only a matter of fineness and of the content of the sodium monoxide (Na<sub>2</sub>O) or its equivalent, which can be from five to ten times the amount contained in high-alkali portland cement, but also a question of the temperature at which the reaction takes place, since this affects reactivity.

The reasons for the strength reductions of portland cement concrete caused by ITC [4] are that in high-heat cements there is more alite  $(C_3S)$  [6, 7], and thus there will be more Ca(OH)<sub>2</sub> in the hydration products. Research [11] with various simulated ITC (Fig. 1) produced the following findings for portland cement mortars:

1. At 20°C the amount of  $Ca(OH)_2$  increased with time, indicating further hydration after eleven days, which was correlated with further strength development.

2. With an increase in ITC, the amount of  $Ca(OH)_2$  increases, but the strength falls. At eleven days there can be as much  $Ca(OH)_2$  present (6.5%) as there is at one year at 20°C.

3. With an increase in ITC, the total amount of  $Ca(OH)_2$  increases.

4. At 20°C the amount of  $Ca(OH)_2$  increases from about 5.5% at eleven days to 6.5% at one year (an increase of 1%), but with increased ITC it drops


FIG. 1—Effect of peak temperature of ITC on the strength and  $Ca(OH)_2$  at eleven days and one year for portland cement [11].

to about 0.5%. This indicates that further hydration has taken place but without improving the strength.

5. The fact that the highest ITC produced not only the lowest strength but also the highest amount of  $Ca(OH)_2$ , in comparison with 20°C curing, might also indicate greater water permeability with increased ITC.

Thus, if these effects are a normal consequence of using high-heat portland cements, the whole premise of relating a low water/cement ratio to low permeability [12] is open to question, particularly when the ITC is large enough to degrade and prevent full strength achievement. The evidence suggests that under the worst conditions the strength is almost half of that stored at  $20^{\circ}$ C. Thus, a paradox is presented to users of concrete: a low W/C which demands the use of high cement contents, is specified to produce concrete of low permeability, but the cement itself prevents this by the conditions it creates for its own hydration. This is a serious matter, and means of correcting this phenomenon urgently need to be considered.

"Good" fly ash pozzolans are not only those that react well at normal temperatures with  $Ca(OH)_2$  but also those that induce water reductions of between 5 and 15%. This fact offers a way of improving concrete by reducing the amount and size of the permeable capillaries [13] and also by producing a greater quantity of calcium silicate hydrate (CSH) gel [14]. With the recognition that under normal circumstances the temperatures involved in quite normal-size elements are in excess of those occurring under standard conditions, the role of fly ash takes on a new significance in terms of *in situ* concrete technology.

On the same basis as previously, Fig. 2 [11] shows the effect of ITC on the strength and Ca(OH)<sub>2</sub> content where 30% of the cement was substituted with a Class F fly ash: the loss on ignition is 3.0%, and the amount retained on a  $45-\mu$ m sieve is 5.8%.

The following can be concluded from Fig. 2:

1. At 20°C and at eleven days, the amount of  $Ca(OH)_2$  present was proportional to the reduced amount of cement present, indicating no reaction; but at one year about 30% of the Ca(OH)<sub>2</sub> had been removed, indicating considerable reaction.

2. At eleven days and as the ITC increased, the strength increased as the  $Ca(OH)_2$  content was reduced.

3. At one year, not only were the strengths significantly greater than those of the 100% portland cement mixtures, but further hydration had taken place over the full range of ITC. The form of this hydration indicates that, with an ITC with a peak temperature of less than  $50^{\circ}$ C, the pozzolanic reaction progressed as normal, but above  $50^{\circ}$ C, it was in some way inhibited because the amount of Ca(OH)<sub>2</sub> increased.

This research, performed with what might be considered a typical British portland cement in terms of a Bogue chemical composition of 56.4% C<sub>3</sub>S (41



FIG. 2—Effect of peak temperature of ITC on the strength and  $Ca(OH)_2$  at eleven days and one year for 70% portland cement and 30% class F fly ash.

cements used by the Cement and Concrete Association from 1965 to 1980 had a mean  $C_3S$  of 56.4% with a standard deviation of 6.5% [15]), independently confirmed the order of strengths found by Bamforth [4]. However, whereas he used concrete, this work was confined to 1:3 cement/standard silica-sand mortar, and it should not be compared with the performance or properties of concrete, particularly regarding critical aspects concerning the permeability, which is sensitive to the pore water volume. (Usually the water content of mortar exceeds 25% volume per volume, whereas in concrete it is below 20% volume per volume).

## **Permeability of Concrete**

## General Considerations

By its very nature, concrete cannot be reduced in scale, mainly because of the effect that aggregate size has on the dilution of cement and water. "Miniatures," such as mortars, therefore should not be related to practical concrete. For instance, there is evidence [16] that, with marginally reactive aggregates, it is possible for expansion to occur with concrete prisms whereas no expansion has been found with mortar bars. In addition, although the strength-related effects of apportioning cementitious material contents and the water-related aspects of slump are meaningful parameters of *in situ* concrete, scaled-down miniatures cannot produce comparable effects.

## Permeability Tests-Discussion of Techniques

The permeability of concrete can be measured indirectly, that is, by neutralization of the surface inward, commonly called carbonation, or directly, either by impregnation of various nonreactant liquids under pressure or vacuum or by water absorption. Whichever method is used, however, there are invariably difficulties of interpretation.

The process of carbonation is protracted, and it may take years for it to be measured effectively, particularly under normal atmospheric conditions. The problems of the interpretation of accelerated carbonation tests under artificial conditions, which alter the products of hydration and the pore-water pressures, are not always appreciated.

Using water to determine permeability presents some difficulties, but under normal atmospheric conditions, it is part of the usual permeability process. The initial surface absorption test (ISAT) [17] is a relatively easy nondestructive test, but it might not properly measure the effect of ITC on the internal permeability of the hardened concrete. Because of this, the modified Figg permeability test [18] was used, as this test measures the rate of water permeability from within the concrete, but it does involve drilling and sealing a cavity 16 mm in diameter by 40 mm deep in hardened concrete.

## The Aggregates

For the small size of specimen used, it is important to take care during the dry drilling operation so as not to disturb the paste-aggregate bond or produce microcracks. Another consideration while drilling the cavity is to avoid local heating, as significant temperature rises, caused by excessive friction, probably change the structure of the hydrated cement gel, both chemically and physically, affecting the area of concrete under testing. The aggregates therefore needed careful selection. These considerations limited the choice of aggregate to a very dense and low-absorption limestone in preference to the more usual siliceous flint or quartzite aggregates, which are very hard and difficult to cut.

It was also important to use aggregates that produced typical water contents that could be related to the slump of the concrete.

The coarse and fine aggregates were selected from a single source of crushed limestone with a water absorption of 0.3% and a density of 2670 kg/m<sup>3</sup>. The gradings of the individual aggregates were as follows:

	Percentage Passing Sieve Size, mm									
	37.5	20	10	5	2.4	1.2	0.6	0.3	0.15	
20-mm coarse aggregate 10-mm coarse aggregate Fine aggregate	100	91 100	2 97	1 4 100	nil nil 97	79	54	31	10	

## The Cement and Fly Ash

The details of the portland cement and fly ash used are given in Table 1.

## The Mixtures

The cement content of structural concrete is normally between 200 and 400 kg/m<sup>3</sup>, and, although the technique for including fly ash varies, it normally requires an increase in the total cementitious material content and a reduction in the water content. The technique [19] used here avoids changes to the aggregates.

The mixtures with and without 25% fly ash, as part of the cement content, and the relationships between the proportions, slump, and cube compressive strength at 28 days are given in Table 2 and in Fig. 3.

## Simulation of the In Situ Temperature Cycle

The previous research [11] had successfully used a technique of several water baths, each held at constant temperature but in steps of 10 degrees Celsius. Heating or cooling the concrete under treatment involved transferring sealed

	Portlan	d Cement	Class F Fly Ash		
Details	Sample Used	British Average Type 1	Sample Used	British Average BS3892 Part 1	
Chemical oxide analysis, %					
Silica, as SiO <sub>2</sub>	20.1	20.5	50.0	50.0	
Insoluble residue	0.6	0.4			
Alumina, as Al <sub>2</sub> O <sub>3</sub>	6.0	5,3	30.4	27.2	
Iron, as Fe <sub>2</sub> O <sub>3</sub>	2.4	2.5	8.9	9.2	
Lime, as CaO	63.6	64.3	2.3	2.5	
Magnesia, as MgO	2.3	1.4	1.5	1.8	
Sodium, as Na <sub>2</sub> O	0.32	0.21	1.1	1.04	
Potassium, as $ar{K}_2O$	0.85	0.70	3.4	2.96	
Sulfate, as SO <sub>3</sub>	2.9	2.6	0.7	0.80	
Loss on ignition	0.6	0.4	3.9	3.2	
Bogue compound composition, %					
C <sub>3</sub> S	51.8	56.4			
$C_2S$	18.6	15.2			
$C_{3}A$	11.0	10.5			
C₄AF	7.3	7.8			
Physical properties					
Color-index (1 to 12)	3	3	3	3.2	
45-μm sieve residue, %	10.7	12.9	7.0	6.5	
Specific surface, m <sup>2</sup> /kg	400	345	355	385	
Density, kg/m <sup>3</sup>	3190	3180	2320	2320	

TABLE 1—Details and information on portland cement and fly ash.

TABLE 2-28-day test results for compressive strength and permeability.

	100% Portland Cement, kg/m <sup>3</sup>			75% I 25% Cla	75% Portland Cement, 25% Class F Fly Ash, kg/m <sup>3</sup>		
	200	300	400	215	320	425	
Slump, mm	70	75	80	70	75	75	
Compressive strength of 100-mm concrete cubes, MPa							
Temperature, 20°C	22.3	42.3	56.0	21.5	43.0	55.7	
Cycle, 40°C	19.6	34.6	43.5	24.3	41.7	51.0	
Cycle, 60°C	18.1	31.9	40.3	26.2	38.1	47.4	
30-minute initial surface absorption test, mL/m <sup>2</sup> /s							
Temperature, 20°C	0.83	0.29	0.18	0.65	0.24	0.16	
Cycle, 40°C	0.90	0.35	0.23	0.47	0.23	0.16	
Cycle, 60°C	1.06	0.42	0.27	0.18	0.15	0.08	
Modified Figg permeability test, s							
Temperature, 20°C	60	350	1100	100	450	850	
Cycle, 40°C	40	190	450	220	450	850	
Cycle, 60°C	20	90	230	310	550	900	



FIG. 3—Chart showing the relationship of proportions to slump and 28-day strength for concrete made with and without fly ash as affected by peak temperature of ITC.

specimens at predetermined times into water baths of different temperatures, as illustrated in Fig. 4.

The problems involved with a greater number of larger specimens caused the work to be confined to a peak profile temperature of  $60^{\circ}$ C but, as the scope was enlarged to test concrete of a wider range of cement contents than previously, the work was considered relevant to the majority of concrete in general use.

#### **Results and Discussion**

The results of the tests are shown in Table 2.

#### Proportions, Slump, and Strength

As shown in Fig. 3 and judged on the basis of 20°C curing, the greater proportions of cementitious material of the fly ash mixtures, coupled with the



FIG. 4-Typical range of in situ temperature cycles (ITC) for structural concrete.

water-reducing effects, produced comparable slump and 28-day compressive strength results over the full range of concretes tested.

However, the effect of the ITC was more pronounced on the portland cement concrete as it significantly reduced the compressive strength, with both increasing ITC and increasing cement content. The effect on the compressive strength of fly ash concrete was less marked, but the ITC nevertheless reduced the strength as the cementitious content increased.

With the information presented in this way (Fig. 3), interpolation is possible for the mixtures; also, the effect of ITC on strength can be more properly assessed. For instance, for concrete subjected to an ITC of  $40^{\circ}$ C, for which 40 MPa *in situ* strength is demanded, the concrete containing portland cement requires 350 kg and the combination requires 239 kg of portland cement and 74 kg of fly ash. However, on the basis of present methods of specification, it would require mixtures containing either 285 kg cement or 236 kg cement plus 73 kg fly ash.

The effect becomes more pronounced as the cementitious material contents alter the ITC. As the difference in *in situ* temperature widens, the size of the ITC becomes considerably smaller for fly ash concrete of nominally the same *in situ* strength; that is, the comparison is between 40°C for the fly ash and  $60^{\circ}$ C for the portland cement concretes. A truer comparison for 40-MPa *in situ* strength would, therefore, exist between mixtures containing 239 kg cement plus 74 kg fly ash (W/C:0.59) and 420 kg portland cement (W/C:0.46). The practical implications of this singular aspect is that, where the effects of thermal cracking have to be minimized, as in water-retaining structures, the amount of crack-distribution steel can be considerably reduced for fly ash concrete.

#### **Permeability Tests**

#### Initial Surface Absorption Tests

The initial surface absorption test (ISAT) is a quick, nondestructive test. However, the results in Fig. 5 show the reversed effects of the ITC on the permeability of concrete with and without fly ash. At 40°C, a concrete made with 200 kg cement and 67.5 kg fly ash (W/C:0.69) has a permeability equivalent to 345 kg cement (W/C:0.56).

This observation can be regarded as putting into question many of the normally accepted methods—such as, for increased durability assume lower permeability by specifying a low W/C.

#### Modified Figg Permeability Test

The modified Figg permeability test is more difficult to perform than the ISAT. Furthermore, a single result ought to be regarded only as indicative. The test requires both more development and more sample points for significant results to be obtained. However, an interpretation of the results is shown in Fig. 6.

On the same basis as before, with an ITC of 40°C, concrete made with 200 kg



FIG. 5—Effect of nominal cement content on the initial surface absorption at 28 days, as affected by peak temperature of ITC.



FIG. 6—Effect of nominal cement content on the Figg permeability at 28 days, as affected by peak temperature of ITC.

of cement and 67.5 kg of fly ash has a permeability close to that for a mixture made with 348 kg of portland cement. The most that can be made of this observation is that it confirms the ISAT result and the two test methods produce a result of the same order.

## Discussion of the Permeability Tests

Recently, details of a test by the International Organization for Standardization (ISO) for determining the permeability of hardened concrete have been made available [20]. However, the method is not sufficiently well developed for standardization: it is assumed that the lack of standard values for the applied pressure will hinder comparison of test results from different sources. Nevertheless, both tests used here confirm that with portland cement and with an increase in ITC the permeability increases, whereas with mixtures containing fly ash, permeability is reduced with an increase of ITC.

It is this reduced permeability that is believed to explain why adding fly ash to concrete reduces or prevents AAR: the alkali-metal ions in the pore fluids of *in situ* concrete are unable to migrate, and so the reaction cannot continue.

## Conclusions

This research shows that the *in situ* temperature cycle markedly increases the permeability of concrete made with portland cement alone. However, incorporating some fly ash as part of the cement reduces the permeability.

In terms of equivalent permeability, Table 3, which is based on Figs. 5 and 6, gives the calculated values of W/C as affected by ITC. These are shown in Fig. 7. Principally, portland cement concrete subjected to an increase of ITC is less resistant to the passage of permeating water. For example, from Fig. 7 a concrete at 20°C with a W/C of 0.50 might have a satisfactory level of impermeability. However, if it is subjected to an ITC of 40°C, its permeability is affected as though it had a W/C of 0.59. If the ITC increases to 60°C, the effect is equivalent to that of a W/C of 0.69.

On the basis of capillary discontinuity, as described by Powers et al [12], this research could change the whole concept of permeability. Concrete made with cement containing at least 25% fly ash and subjected to an ITC of more than 40°C is equivalent in permeability to a portland cement concrete with a W/C of less than 0.42 at 20°C.

Because the whole basis for concrete impermeability is traditionally related to W/C, the early-age temperature cycles require urgent consideration. This is particularly true for concretes made with high-heat portland cements. It is also clear that, where permeability is a limited state consideration, concrete is placed at unnecessary risk if it does not include an effective proportion of fly ash, pozzolan, or similar material.

	Water/ Cement	Permeability in Terms of Water-Cement Ratio of Temperature- Affected Concrete at Peak Temperature of ITC							
Rat Stand Cement Cur Mix 20°	Standard	40°C			60°C				
	Cure, 20°C	ISAT	Figg	Ā	ISAT	Figg	Ā		
100% Type I	0.45	0.54	0.55	0.55	0.59	0.67	0.63		
portland	0.50	0.58	0.60	0.59	0.64	0.74	0.69		
cement	0.60	0.66	0.71	0.69	0.71	0.85	0.77		
	0.70	0.75	0.80	0.78	0.80	0.93	0.86		
	0.80	0.85	0.89	0.87	0.90	1.00	0.95		
75% Type I	0.45	0.40	0.46	0.43	0.35	0.43	0.39		
portland	0.50	0.45	0.49	0.47	0.37	0.46	0.42		
cement,	0.60	0.53	0.56	0.55	0.40	0.51	0.46		
25% Class	0.70	0.62	0.62	0.62	0.46	0.55	0.51		
F Fly Ash	0.80	0.68	0.68	0.68	0.51	0.59	0.55		

 TABLE 3—Changes in permeability, expressed in terms of equivalent water/cement ratio, as affected by the peak temperature of ITC (Interpolated from Figs. 5 and 6).



FIG. 7—Permeability expressed in terms of water/cement ratio as an effect altered by the peak temperature of the ITC with and without 25% fly ash as part of the cement content of concrete.

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# Effects of Early Heat of Hydration and Exposure to Elevated Temperatures on Properties of Mortars and Pastes with Slag Cement

**REFERENCE:** Roy, D. M., White, E. L., and Nakagawa, Z., "Effects of Early Heat of Hydration and Exposure to Elevated Temperatures on Properties of Mortars and Pastes with Slag Cement," *Temperature Effects on Concrete. ASTM STP 858*, T. R. Naik, Ed., American Society for Testing and Materials, Philadelphia, 1985, pp. 150-167.

**ABSTRACT:** The behavior of slag cements combining substantial amounts of separately ground granulated blast-furnace slag is compared with that of portland cements. The effects on mortars and pastes of exposure to elevated temperatures during early-stage hydration and at later ages has been determined. Compressive strength, density, micro-structure, permeability, and dimensional change were the primary properties investigated. The effects of long-term exposure at temperatures up to 250°C were evaluated. Very low permeabilities,  $< 10^{-8}$  darcys ( $\mu$ m<sup>2</sup>), were maintained in most of the mortars. The phase changes with time were determined by X-ray diffraction.

Compressive strengths up to 200 MPa and higher were found in some of the mortars. Pore structures by mercury porosimetry were also investigated, revealing very fine pore structures and some changes with elevated temperature.

**KEY WORDS:** Slag cements, compressive strength, blended cements, permeability, silica fume, viscosity, granulated slag, pore structure, high temperature, porosity, longterm exposure, phase changes, hydrated pastes, density, mortars, concrete

This study was part of a research effort on the development of mortars and pastes to optimize particular physical properties at temperatures ranging from 27 to 250°C. In many cementitious mixtures industrial by-products, such as silica fume and ground-granulated blast furnace slag, have been used

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successfully [1-6]. One method is to substitute these by-products partially for the more expensive or more energy-consuming portland cements in proposed mixtures. Such materials are usually classified as mineral admixtures, but when used in quantities up to 60 to 80% of the total cementitious component, as is frequently done with glassy slag, these materials are more properly called "cements." While comparing the physical properties of some of these new mixtures, we have found that mixtures containing different proportions of these by-products have, in a number of cases, produced a more favorable product. They have the further advantage that they modify the composition of the cementitious component to approach more closely that of natural silicate rocks with which they may be used [7].

The objectives of this study on slag cements exposed to high temperatures were the following:

(a) to attempt to prepare formulations which would perform well over a broad temperature range at temperatures up to well above  $100^{\circ}$ C,

(b) to investigate the heats of hydration and structural development of slag cements during the very early and later stages of hydration using three different slag compositions on five different mixtures,

(c) to measure the pore structure dependence on temperatures of 27, 38, 60, and  $90^{\circ}$ C at pressures up to 410 MPa (60 000 psi),

(d) to investigate the long-term exposure to temperatures up to  $250^{\circ}$ C for curing times up to 28 days, and

(e) to compare the physical properties of strength, density, porosity, and heats of hydration for both the different proportions of slag versus cement and also the pure cement mixtures.

Several mixtures had favorable physical properties after curing. A mortar containing a 14% slag/23% portland cement mixture, which was continuously cured at temperatures from 23 to 250°C, was the most dense, had the highest compressive strength, and had favorable pumpability properties.

## **Sample Compositions**

Five mixtures containing granulated blast furnace slag and portland cement were altered in composition by the addition of quartz, silica fume, and gypseal (CaSO<sub>4</sub> $\cdot$ 0.5H<sub>2</sub>O) to determine the optimum calcium oxide/silicon dioxide (CaO/SiO<sub>2</sub>) ratio for a mortar mixture to perform at ordinary and elevated temperatures. The compositions of the five mixtures proposed are given in Tables 1 and 2. Table 1 lists the type of material as a percentage of the total mix, and Table 2 lists the total chemical compositions of the fineparticle (cement-size) components of these mixtures. An American Petroleum Institute (API) Class C cement was used in the first mixture; for the others, API Class H cement was used. The Class C cement was equivalent to a sulfate-resistant ASTM Type III cement, while the Class H cement had more

	Cement/Slag Mixtures							
Material	79-050	81-050	81-121	82-11	83-01			
Cement	24.24	36.29	33.14	23.73	23.73			
Slag	24.24	8.29	12.81	14.24	14.24			
Ouartz	20.48			18.98	18.98			
Silica fume		8.34	4.30					
Gypseal <sup>a</sup>		2.22	1.15					
Sand <sup>b</sup>		27.16	33.44	28.47	28.47			
Admixture <sup>c</sup>		1.15	1.02	0.57	0.57			
Water	31.03	16.55	14.14	14.01	14.01			
Defoamer	•••			0.01	0.01			
$W/C^d$	0.64	0.29	0.27	0.36	0.36			
W/S <sup>e</sup>	0.45	0.29	0.27	0.24	0.24			
Cement type	Class C		——————————————————————————————————————	ss H				
Slag type	~	— B19 —	>>	<b>B</b> 49	B66			

 
 TABLE 1—Materials used in five slag/cement compositions as a percentage of the total mixture.

"Calcium sulfate hemihydrate.

<sup>b</sup>ASTM Test for Compressive Strength Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens) (C 109-80) sand.

<sup>c</sup>Superplasticizers.

 $^{d}W/C = water/(cement + slag + silica fume + gypseal + admixture).$ 

W/S = water/(components in Footnote c) + fine quartz.

 $SiO_2$ , alumina (Al<sub>2</sub>O<sub>3</sub>), and titanium dioxide (TiO<sub>2</sub>) and less ferric oxide (Fe<sub>2</sub>O<sub>3</sub>) than the Class C cement. The same granulated blast furnace slag composition (MRL No. B19) was used for Mixtures 79-050, 81-050, and 81-121; for Mixtures 82-11 and 83-01, the slag compositions were similar, as can be seen in Table 2. All were glassy, containing >95% glass. For Mixtures 81-050 and 81-121, the silica fume (ferrosilicon dust) was reduced from 8 to 4% while the blast furnace slag was increased from 8 to 12%, maintaining approximately 15% total for the fume/slag addition. The addition of 14% slag to the last two mixtures, 82-11 and 83-01, was chosen both to maintain a relatively constant Al<sub>2</sub>O<sub>3</sub> and magnesium oxide (MgO) content for the "fine solids" in all five comparative mixtures and to formulate an optimum CaO/SiO<sub>2</sub> ratio. The contributions of the slags can be seen by comparing the slag chemical compositions, given in Table 3.

## **Experimental Procedures**

#### Calorimetry

Early heat of hydration was determined with a Thermonetics Seebeck-type isothermal calorimeter, Model C-12-45-2G [8]. A 3-g sample of each of the

Chemical		Cement/Slag Mixtures						
	79-050	81-050	81-121	82-11	83-01			
SiO <sub>2</sub>	48.50	29.78	29.24	50.54	50.67			
$Al_2O_3$	4.19	4.33	4.33	4.57	4.04			
TiO <sub>2</sub>	0.21	0.22	0.22	0.41	0.20			
$Fe_2O_3$	1.80	2.83	2.83	1.81	1.81			
MgO	4.47	4.73	4.72	3.30	4.46			
CaO	37.83	52.29	52.29	37.01	36.40			
MnO	0.10	0.08	0.08	0.12	0.14			
SrO	0.04	0.05	0.05		0.04			
BaO	0.01	0.01	0.01		0.02			
Na <sub>2</sub> O	0.13	0.21	0.20	0.14	0.10			
K₂Ō	0.31	0.51	0.50	0.38	0.33			
P <sub>2</sub> O <sub>5</sub>	0.04	0.06	0.06	0.04	0.04			
SO <sub>3</sub>	1.76	3.50	3.50	1.49	0.80			

 
 TABLE 2—Chemical composition of the fine powder components for five slag/cement mixtures given as a percentage of the total.<sup>a</sup>

"Sand and water are not included in the total composition.

 
 TABLE 3—Chemical composition of three different slags used in the slag/cement mixtures, given in percentages.

	MRL Sample No.					
Chemical	<b>B</b> 19	<b>B</b> 49	<b>B</b> 66			
SiO <sub>2</sub>	32.7	33.4	34.3			
$Al_2O_3$	8.3	12.3	10.2			
TiO <sub>2</sub>	0.4	1.3	0.5			
$Fe_2O_3$	1.2	0.7	0.7			
MgO	8.9	6.7	11.4			
CaO	44.6	43.1	40.6			
MnO	0.3	0.4	0.5			
SrO	0.06		0.05			
Na <sub>2</sub> O	0.25	0.34	0.19			
K <sub>2</sub> O	0.34	0.63	0.45			
$SO_3^a$	3.1	2.8				
$S^a$		• • •	1.31			
Blaine specific surface area, cm <sup>2</sup> g <sup>-1</sup>	5590	4530	5550			

<sup>a</sup>Sulfur contents similar; one was reported as sulfur, two as SO<sub>3</sub>.

mixtures was taken from a larger mixture and placed into the calorimeter chamber. A piece of filter paper with five holes in it was cut to fit over the sample to ensure uniform distribution of the water over the sample. A syringe was wetted with deionized water, filled to maintain the water/cement ratio (W/C) given for each of the mixtures, and then put into the proper calorimeter recess and allowed to come to thermal equilibrium with the sample before

injection. The heat of hydration was followed on a strip chart recorder immediately after the water was injected through a hole in the cover of the calorimeter. For an accurate representation of the first peak, the chart speed was increased to 1 cm/min, and the maximum heat of hydration was reduced to allow full-scale response for these mixtures (full scale was set at 2mV/100divisions). Full-scale response for portland cements was 5 mV/100 divisions. For the second and third peaks, the chart speed was set at 1 cm/h, and the maximum heat of hydration was set for full-scale reading of 0.5 mV/100 divisions. The results are the average of three replicate measurements. For some of these tests, the second derivatives of the heat of hydration curves were plotted to obtain greater detail in early-stage hydration (second and third peaks) [8,9].

## Mercury Porosimetry

Porosity and pore size distribution measurements were made on a Quantachrome Autoscan Porosimeter Model SP-200, which has sample cells of 3 and 7 mL. The stem volumes correspond to intrudable volumes of slightly over 0.5 and 2.0 mL when filled with mercury for the smaller and larger cells, respectively. This scanning porosimeter produces a continuous recording of the intrusion or extrusion of mercury into the cell, therefore, all the small inflection plateaus or steps were recorded. The small cell was used for most measurements to minimize the quantity of mercury in the cell. Measurements may be made in either the linear mode or the log mode. Using the latter mode allows the plotting operation of pore volumes versus the pore radius directly. The Washburn equation

$$P = -2\gamma \cos \theta / rK$$

was used to calculate pore volumes, V and dV/dP as a function of the radius [this function is equivalent to  $dV/d(\log r)$ , where r = radius of pores, P is intrusion pressure, and K = instrument constant], and  $\theta =$  wetting angle for mercury (assuming  $\theta = 140^{\circ}$ ), and  $\gamma =$  surface tension. Most of the water was extracted from the samples before testing by immersing them in acetone for 24 h, then putting them into an ether environment, and then drying them in a vacuum furnace at room temperature (23°C) each for 24 h [1].

#### Viscosity

The rheological properties of each of these mixtures were determined to produce pumpable slurries or workable mortars. The slurries were designed to be pumpable for up to 2 h with a viscosity of less than 2000 cP (2 Pa  $\cdot$  s), and the mortars were to be workable for up to 3 h with a viscosity of less than 30 000 cP (30 Pa  $\cdot$  s) after 2 h. The measurement of viscosity has been shown

to be one of the more critical measurements of the structural changes in the early stages of hydration because of changes in composition [10]. All the measurements were made using a Haake Rotovisco RV3 viscometer with the MK50 or MK500 measuring heads. Serrated rotor and measuring cup sensor systems, SVIIP and MVIIP, were used with these cementitious mixtures in order to ensure homogeneous flow within the mixtures when they were being tested. All the slurries and mortars were prepared according to ASTM Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency (C 305-82). Testing was done within a constant temperature chamber at  $38^{\circ}C \pm 0.01^{\circ}C$ . A 200-mL sample was prepared and then tested at 30 min after the initial mixing.

#### Permeability

Permeabilities are among the most important characteristics of materials that may be used for sealing purposes as, for example, nuclear waste isolation applications [7,11]. For this reason a measuring apparatus was constructed of a miscellaneous Grade-300 series austenitic stainless steel which would measure water permeability directly up to driving pressures of 34.5 MPa (5000 psi) (factor of safety = 4). This system, shown in Fig. 1, utilizes a commercial nitrogen gas cylinder and a regulator with a Hooke valve as a source of stable pressure, generally maintained at 4 to 5 MPa (600 to 700 psi) for this set of experiments. Samples with Mixture 79-050 used a driving pressure of 7 MPa (1000 psi). The gas pressure drives a piston which forces deionized water through the sample. The samples were sealed into brass rings with Aqutapoxy, which is lime [Ca(OH)<sub>2</sub>]-resistant, since most cementitious samples are cured in a saturated Ca(OH)<sub>2</sub> medium. Other epoxy products were unstable in the  $Ca(OH)_2$  medium. The cylindrical samples, approximately 25.4 mm in diameter and 10 mm thick, were sealed with an O-ring to prevent leakage of water around the brass ring. A porous plate of Type 316 stainless steel, 25.4 mm in diameter, and 3.18 mm (1/8 in.) thick with 40-µm holes supported the sample over the liquid drainways. The measured rate of water flowing through the sample, in millilitres per second, was used to calculate the water permeability directly. Plexiglas seal chambers were built around each cell to decrease the amount of evaporation during testing. Load cells, which were used to convert the weight of water to an electrical signal for the recorder, have capacities of 50, 100, and 500 g for different sensitivities of measurements.

A dimensionless Reynolds number established that the flow during testing was within the laminar flow regime (viscous forces predominate over the inertial forces). The Reynolds numbers for flow through the cured cement paste and mortar samples were in the range of  $10^{-7}$  [11] using a 20 to 200-Å (2 to 20-nm) mean cementitious particle size. In that study the highest frequency

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FIG. 1-Schematic drawing of permeability apparatus.

pore diameter of 20 to 200 Å (2 to 20 nm) was used as a characteristic length to give a Reynolds number of

$$N_R = 5 \times 10^{-8}$$
 to  $5 \times 10^{-7}$ 

The intrinsic permeability could also be interpreted as a unique pore area governing the flow; when this property of the porous medium was used as a characteristic length, the Reynolds number was

$$N_R = 2.5 \times 10^{-7}$$

It is significant that even with driving pressures of up to 27.6 MPa (4000 psi), the flow regime continued within the laminar region. These data are based on

the theoretical maximum specific discharge for which Darcy's law holds developed by Hubbert [12] on flow-through porous media. Using Hubbert's criteria, flow is within the laminar regime up to velocities through the samples of 200 m/s. The experimental tests were performed at much lower velocities than that.

The water-cured samples were prepared for testing by surface drying them, then epoxy-sealing them into brass rings, and then returning them to a greater than 95% relative humidity chamber for 24 h or longer so they remained within the saturated condition up to and continuing through the testing period.

## Results

#### Heat of Hydration

The rates of heat generated during the very early stages of hydration (less than 10 min) as well as in later stages of hydration (during the next 30 h) were compared with both pure portland cement and each of the other blended granulated slag cements. For these sets of experiments, the mixtures were hydrated at 38°C for at least 60 h.

The first thermal peak, which occurs during the first 10 min of hydration, was reduced by approximately 40 to 140% when the CaO/SiO<sub>2</sub> ratio was reversed from about 50:30 (in Mixtures 81-050 and 81-121) to about 30:50 (in Mixtures 82-11 and 83-01), as shown in Fig. 2.

The second peak is a single peak for the pure portland cement, while in the slag/cement mixtures it is resolved into two peaks, depending on the temperature, slag content, and slag characteristics, including the specific surface area [3, 9]. The two peaks were not resolved for the 50:30 81-050 and 81-121 mixtures. In earlier studies [3, 9] these peaks were shown to be distinct by differentiating the rates of heat liberated (that is, producing a second derivative plot); these were also found to be more pronounced at the 38°C curing condition. In the earlier studies with a single slag, the time of the third peak was found to be independent of the slag content (in the range 60:40 to 35:63 for cement/slag mixtures); however, the height of the third peak was found to be dependent on the slag content. As can be seen in Table 2, the CaO/SiO<sub>2</sub> content remained within the 30:50 range for Mixture 79-050 as well as for the 82-11 and 83-01 mixtures. In this study the reduction of the CaO content of the fine (cement-size) powder from that in the 50:30 mixtures had slowed the hydration time for the third peak from 10 h to approximately 18 h. The additional factor, the presence of very fine silica fume in the 50:30 mixture samples and not the 30:50 samples, probably also contributed to the acceleration of the hydration in the former.

For many projects that require placing large masses of concrete, the lag in heat generated is a significant positive property for these mixtures. This has



FIG. 2—Heat of hydration for slag/cement mixtures.

been discussed in detail elsewhere [13]. When these were compared with the heat generated from a pure portland cement paste, as shown in Fig. 2, the reduction in the peak rate of heat generation is more significant.

#### Viscosity

Quantitative rheological measurements on three slag/cement mortar mixtures (83-01, 81-121, and 81-050) and the slag/cement slurry mixture, 79-050, showed that the 50:30 CaO/SiO<sub>2</sub> mixtures (81-050 and 81-121) containing condensed silica fume, a fine powder, had the lowest viscosities. They also had the highest superplasticizer content to aid in fluidifying. In addition, the viscosity at the lower shear rates (less than 25 s<sup>-1</sup>) increased in the mixtures containing  $5-\mu m$  quartz and no silica fume, which also contained increased amounts of slag. The viscosities increased with a decreasing water/ total fine solids ratio (W/S), from 0.24 to 0.27 to 0.29 in the superplasticized mixes (81-050, 81-121, and 83-01). The shape, magnitude, and direction of the shear stress/shear rate loops in Fig. 3 indicate the nature of the structural changes in the slurry during testing. Except for Mixture 83-01, there were only minimal changes in the structural habit of all these mixtures. The structural loop given for Mixture 83-01 has the general characteristics typical for pseudothixotropic flow within a mixture. The Mixture 79-050, having no superplasticizer, exhibited a significant yield stress. The effects of superplasticizers on dispersion phenomena and rheological characteristics in such blended cements have been discussed elsewhere [14-16].

## Pore Structure

The cumulative pore volume of blends of ground-granulated blast furnace slag mixed with cement were measured by intruding mercury into the pores at pressures up to 400 MPa (60 000 psi). Roy and Parker [1] found a finer pore structure in the slag cement paste, as well as a lower total cumulative pore volume (porosity), in comparison with a pure portland cement paste under identical conditions, as shown in Fig. 4. They related the fine pore structure to a fine microstructure, visible in the scanning electron microscope (SEM).



FIG. 3-Rheological properties of slag/cement mixtures.



FIG. 4—A comparison of cumulative pore volume for pastes of a pure portland cement and a 40:60 (weight proportion) cement/slag mixture at intrusion pressures up to 400 MPa (60 000 psi).

The differential pore radius of mixtures with portland and slag cements are compared in Fig. 5. Contrasted are 0.40 W/C pastes cured at 45°C, which show that the maximum in the differential plot (the critical pore radius) occurs at a considerably smaller pore size than with the pure portland paste. The  $30:50 \text{ CaO/SiO}_2$  mixture (83-01) was compared with both a 50:30 CaO/SiO<sub>2</sub> mixture of 60% slag to 40% cement and a pure portland cement. Both the pure portland cement and the 60:40 slag/cement mixtures had a higher pore volume than the 83-01 mixture at a given intrusion pressure. The crosshatched portion in Fig. 5 shows the range in data for curing times of 7 to 28 days and in temperatures from 23 to 90°C. The smaller pore volume of Mixture 83-01 samples was partly due to the quartz powder and sand content. These samples also had a finer pore structure than the pure portland paste.



FIG. 5—A comparison of differential pore radius for a pure portland cement, a 60:40 (weight proportion) slag/cement mixture, and Mixture 83-01.

The pore volume relationship with intrusion pressure showed a decrease in pore volume of approximately 50% when the samples were cured for 3 to 28 days, respectively, at each curing temperature. Based on the total cumulative pore volume of pieces broken from a 2.54 by 5.1-cm (1 by 2-in.) high cylindrical sample, typical non-slag-containing samples developed fractional pore volumes as low as 0.15 mL per millilitre of sample after 56 days of curing at 90°C. The 83-01 mixture achieved a pore volume of 0.15 after only 3 days of curing at 90°C. After 28 days of curing, this mixture had a pore volume of 0.07 mL per millilitre of sample, a very low pore volume for a cementitious mixture (see Fig. 6).

#### Permeability

The intrinsic permeability for each of these mixtures was less than  $10^{-8}$  darcys ( $\mu$ m<sup>2</sup>), as can be seen in Table 4. The exception was two sets of measurements using Mixture 81-121 at 60 and 90°C for 56 and 90 days curing time. Since these mixtures were impermeable ( $<10^{-8}$  darcys) after 180 days of curing at these temperatures, one can assume that these mixtures have equivalent permeabilities to those of dense granitic and basaltic rocks. The high temperature data from Mixture 82-11, with a similar composition to that for 83-01, also showed that the mixture remains nonpermeable up to 250°C curing temperature.

The porosities/permeabilities of these mixtures are comparable to those for bentonites used for backfill barrier components. Nowak [17] estimated



FIG. 6—The pore volume versus intrusion pressure relationships for Mixture 83-01, which was cured for 3 and 28 days.



TABLE 4—Water permeability of slag/cement mixtures.

 $^{a}1 \text{ psi} = \sim 6.89 \text{ kPa}.$ 

breakthrough times of  $10^3$  to  $10^4$  years for the bentonites with effective porosities from 0.01 to 0.1, bulk densities of approximately 2 g/cm<sup>3</sup>, and interstitial groundwater velocities from 0.03 to 30.5 m/year (0.1 to 1000 ft/year). Though the materials are not strictly comparable, the current results suggest that these mixtures would have similar very long breakthrough times.

## Compressive Strength and Bulk Density

The effects of longer term exposure at high temperatures ranging from 23 to 90, 175, and 250°C were investigated. The compressive strength of Mixture

82-11, the 30:50 CaO/SiO<sub>2</sub> high-temperature-cured samples, was the highest (245 MPa), as shown in the left half of Fig. 7. A comparison between these slag/cement mixtures revealed that the 14% slag compositions, Mixtures 83-01 and 82-11, had compressive strengths about 25% higher than or equal to those for the other mixtures, for example, the average for the 27°C-cured samples was 75 MPa; the average for the 27°C-cured 83-01 samples was approximately 100 MPa after 28 days of curing and approximately 225 MPa for the 175°C-cured samples, increasing only slightly between 7 and 28 days of curing. Following the same pattern, the 90°C-cured samples, excluding the 30:50 mixtures, had average strengths of 70 to 111 MPa, whereas the 83-01 and 82-11 samples had average compressive strengths of 125 and 175 MPa after 28 days of curing at 90 and 250°C, respectively. Generally, the higher temperature curing enhanced the strength, as well as the Young's modulus. Presumably it would enhance other properties such as thermal conductivity, which depend on factors similar to those controlling the modulus [18].

The bulk density for the 50:30 CaO/SiO<sub>2</sub> mixture (Fig. 8) decreased with curing time, whereas that for the 30:50 mixtures of 83-01 increased to 2.2 and 2.3 g/cm<sup>3</sup> for the 23 and 90°C-cured samples, respectively. The high-temperature form of the 30:50 mixture had a constant 2.2-g/cm<sup>3</sup> density throughout the curing times up to 28 days.

## **Reaction Products**

The cured slag/cement mixtures contained some residual cement phases [including  $\beta$ -Ca<sub>2</sub>SiOH ( $\beta$ -C<sub>2</sub>S)] and quartz, plus the reaction products ettringite, calcium hydroxide, monosulfate, and amorphous calcium silicate



FIG. 7-Compressive strength of slag/cement mixtures.



FIG. 8-Bulk density of slag/cement compositions.

hydrate (C-S-H), the latter becoming better crystallized with higher temperatures. The occurrence of calcium hydroxide as an end product in the samples was coincident generally with lower strength in the samples; it no longer appeared as a stable end product after the higher temperature curing or longer curing times for the 79-050, 81-050, and 81-121 materials. Calcium hydroxide did not occur as an end product even at lower temperatures within the higher strength mixture, 83-01 (at 28 days), as can be seen in Table 5.

## **Discussion and Conclusions**

Blends of ground-granulated blast furnace slag with portland cement have been used to prepare pastes and mortars with unusually high strengths and other favorable properties even at temperatures up to  $250^{\circ}$ C. The use of such blends also has great potential for optimal utilization of industrial by-products and for saving energy. It has been estimated that an energy savings of 1.7 GJ of energy per 1000 kg of cement could be achieved by using slag cement containing 65% slag rather than normal portland cement [9]. The strengths of Mixtures 81-050 and 81-121 are illustrated in Fig. 9. In this figure, the high-temperature slag/cement mixture, 82-11, and its counterpart low-temperature mixture, 83-01, utilize the minimum amount of cement with the highest compressive strengths.

These two mixtures were shown to possess the most favorable physical properties of strength, durability, pumpability, and low porosity. They contain 23% cement and 14% slag, plus other components including fine silica, sand, and water. The chemical composition (of fine, cement-size particles) is maintained at about 50% silica and approximately 30% CaO. These mixtures have high compressive strengths, correspondingly high Young's moduli, and very low porosities with most of the pore volume occurring at pore radii below about 40 Å. The cumulative pore volume decreases with curing time







FIG. 9—A comparison between the amount of cement within the slag/cement mixtures and their compressive strength properties.

and with curing temperature. The mixtures have extremely low permeabilities ( $<10^{-8}$  darcys); they have a low heat of hydration while developing very good (35-MPa) strengths even at three days at 27°C. The workability and pumpability of each of these mixtures had been determined, and the 83-01 mixture was workable for up to 3 h after the dry and liquid components were initially mixed. The properties of the slag/silica fume combinations were less fully explored, but they also show considerable promise.

There are significant implications for durability from the preparation of strong cementitious materials with dense microstructures and very low permeabilities produced from the same slag-rich compositions over a broad temperature range from room temperature to 250°C. The accelerating effect of temperature is partly a substitute for time when projecting longer term durability [7]. Hence, such materials show high promise of long-term durability, since their properties show continuity irrespective of temperature.

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## The Willow Island Collapse: A Maturity Case Study

**REFERENCE:** Halvorsen, G. T. and Farahmandnia, A., "The Willow Island Collapse: A Maturity Case Study," *Temperature Effects on Concrete, ASTM STP 858*, T. R. Naik, Ed., American Society for Testing and Materials, Philadelphia, 1985, pp. 168-176.

**ABSTRACT:** The Willow Island cooling tower collapse of April 1978 is reviewed with respect to the development of concrete maturity during construction. The tower shell was constructed during the fall and spring prior to the collapse. Fall and spring weather conditions were similar with regard to the potential for concrete strength development. However, an increased pace of construction in the spring effectively decreased the maturity of the concrete at the time it was subjected to construction loads. More than half of the concrete lifts placed during the spring construction of the cooling tower shell were supported by concrete with a maturity similar to that of the concrete in Lift 28, which failed during the placement of Lift 29. The collapse might have occurred sooner if several days of rainy weather had not apparently delayed the construction.

**KEY WORDS:** collapse, concrete, reinforced concrete, concrete strength, construction, cooling towers, failure, maturity

The April 1978 partial collapse of a power plant cooling tower under construction near Willow Island, West Virginia, has received much attention in the technical and popular press because of the particularly large loss of human life. The public reports of this collapse have generally focused on the trigger mechanism for the collapse [1-3], although a number of other questions have been raised [4].

At West Virginia University, Morgantown, West Virginia, some additional study of the collapse has been conducted as unsponsored research, based solely on data available within the public domain [5]. This paper reports the details of that work. The original intention of the study was to use the maturity concept, along with field concrete strength data, to describe the structural capac-

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ity of the tower at various times, to perform structural analyses of its partially completed shell, and, finally, to determine the variation in the safety margin during the course of construction. This proved too ambitious because of the limited amount of field strength data available in the open literature. Although these goals could not be achieved fully, this study still provides some useful information about the Willow Island collapse.

## The Maturity Concept

The maturity concept is a useful tool for estimating the strength of concrete during construction. This concept dates back to work by Saul [6] and Plowman [7] and has been the object of much recent interest [8]. In the United States the maturity concept has been extensively applied to the investigation of failure of concrete structures since the early 1970s [1-3, 9-11].

The concept of maturity provides a single parameter which accounts for the effects of curing time and curing temperature. The most common definition of maturity is

$$M = \int_{0}^{t_{1}} [T(t) - T_{o}] dt$$
 (1)

where

- M = concrete maturity at Time  $t_1$  (in degree-hours or degree-days),
- T(t) = concrete temperature at any time, t, in degrees,
  - $T_o =$  lowest temperature at which concrete gains strength, and
  - $t_1 =$  time since water was added to the cement (typically in hours or days).

The value of the datum temperature to be used in Eq 1 has been estimated differently by various researchers; as reported in Ref 12, most indicate that it is in the range of -10 to  $-12^{\circ}C$  (11 to  $14^{\circ}F$ ). At present, the datum temperature used most commonly in North America is  $-10^{\circ}C$  ( $14^{\circ}F$ ).

Maturity may be used to infer strength levels through the use of a strengthmaturity relationship or may be used directly as a material strength index. If the strength-maturity relationship is used to assist in establishing a construction schedule, for example, it is absolutely essential that this relationship be obtained through a process of pretesting the actual mix constituents that will be used in the field. In the case of a failure investigation, it is also highly desirable to determine a strength-maturity relationship from tests of concretes made with the same constituent materials, as has been done in Refs 1 and 11. If this is impossible, the investigator might turn to general relationships, such as those determined by Lew and Reichard [12] through a regression analysis of large amounts of strength-maturity data.

#### **Maturity Analysis**

To evaluate the development of maturity in a given application, the temperature history of the concrete must be known. Since information on *in situ* concrete temperatures is seldom available after the fact, investigators have normally approximated the temperature of concrete in a partially completed structure as being the same as the temperature of the surrounding air [1, 11]. For a particular construction project, even the job site records may not provide this information in sufficient detail, so it is necessary to go some distance from the site to obtain detailed, reliable temperature information. The present study used the average daily temperature data recorded at the Parkersburg, West Virginia, office of the National Weather Service [13]. This weather station is located about 24 km (15 miles) from the Willow Island site.

Construction of the cooling tower shell was initiated on 10 Oct. 1977, halted for the winter on 16 Nov. with the placement of Lift 10, and resumed on 27 March 1978. The collapse occurred on 27 April 1978, during the placement of concrete for Lift 29. The official investigations of the collapse indicate that failure was initiated in Lift 28 as a result of a load combination dominated by the cathead gantry crane system which hoisted materials to the work platform area [1-3].

For reference purposes, Fig. 1 illustrates the average daily temperature during the construction of the cooling tower shell. For the time axis of this figure, Day 0 corresponds to the first day that concrete was placed in the tower shell; Day 200 is the day following the collapse.

Using the average daily temperature data and the definition of maturity, it is possible to construct a "maturity history" for the tower shell concrete, as



FIG. 1-Average daily temperatures during the construction of the Willow Island cooling tower.

shown in Fig. 2. This is identical to a representation of maturity development for the concrete in Lift 1 of the shell. This relationship indicates the potential for concrete curing and strength development.

Figure 3 illustrates the development of maturity at an expanded scale for periods of fall and spring construction, respectively. The two curves have virtually the same slope. This indicates a similar development of maturity, and consequently a similar potential for concrete strength development.

The pace of construction is an important factor to introduce at this point. Significant construction data extracted from Ref 4 are reproduced in Table 1. The tower shell concrete was placed in lifts 1.25 m (5 ft) high. As indicated in



FIG. 2-Maturity history for cooling tower shell concrete, indicated in degree-days.



FIG. 3-Maturity history during the fall and spring construction, indicated in degree-days.

Lift	Day	Date	Lift	Day	Date
1	М	10/10/77	14	Th	3/30/78
2	Μ	10/17/77	15	F	3/31/78
3	Th	10/20/77	16	М	4/3/78
4	Μ	10/24/77	17	Т	4/4/78
5	F	10/28/77	18	W	4/5/78
	М	10/31/77	19	F	4/7/78
6	Т	11/1/77	20	Μ	4/10/78
	W	11/2/77	21	Т	4/11/78
7	Th	11/3/77	22	W	4/12/78
	F	11/4/77	23	Th	4/13/78
8	Т	11/8/77	24	F	4/14/78
9	М	11/14/77	25	Μ	4/17/78
10	W	11/16/77	26	W	4/19/78
11	М	3/27/78	27	М	4/24/78
12	Т	3/28/78	28	W	4/26/78
13	w	3/29/78	29	Th	4/27/78

 
 TABLE 1—Concreting schedule for cooling tower shell (after governor's commission on Willow Island [4]).

Table 1, most of the lifts were placed in a single workday. During the fall construction, 10 lifts of the shell were placed during a 37-day period, while in the spring 18 lifts were placed in 30 days. The effect of this increase on the pace of construction is significant in terms of the rate of maturity and, thus, of the concrete strength development.

Figure 4 compares the potential development of maturity during the fall and spring construction if the construction of each lift is considered to be a unit of time. Separate curves are indicated for Lifts 1 through 10, the fall construction, and Lifts 11 through 20 and 21 through 29 in the spring. The curves are replotted in Fig. 5 to an expanded vertical scale and to a common point of beginning to emphasize the differences in the slope of the curves. The slope of the curve for Lifts 1 through 10 is significantly steeper than the curves for Lifts 11 through 20 and 21 through 29, a consequence of the increased speed of construction in the spring.

The conclusions that may result from a study of Fig. 5 are more readily apparent if the data are analyzed to represent the maturity of concrete immediately supporting construction of a new lift in the shell. Figure 6 indicates the maturity of concrete in the previous lift, at the time a particular lift is placed. Although the design of the scaffold system may have intended that load transfer take place over the two previous lifts, analysis reported by Lew and Fattal [3] indicates that this did not occur. Thus, the capacity of the lift immediately below that being placed is a key factor in the collapse mechanism.

The data of Fig. 6 express a material strength index for the concrete supporting any freshly placed lift. If a well-defined strength-maturity relationship was available, an evaluation of material and structural capacity would be feasi-



FIG. 4—Maturity development for Lifts 1 through 29, indicated in degree-days.



FIG. 5-Comparison of maturity by lifts, indicated in degree-days.

ble. However, the available strength data in this case are quite limited. Lew et al [I] report compressive strength-maturity relationships obtained from both field tests conducted during the tower construction at Willow Island and laboratory studies performed on concrete made with the same constituent materials during investigation of the failure. The field cylinder test data indicate that a strength of 5 MPa (725 psi) is attained at a maturity of approximately 50 Celsius degree-days (90 Fahrenheit degree-days), while a strength of 10 MPa (1450 psi) is attained at approximately 70 Celsius degree-days (125 Fahrenheit degree-days. The maturity of Lift 28 at failure is estimated to be about 24 Cel-


FIG. 6-Maturity of the previous lift, indicated in degree-days.

sius degree-days (43 Fahrenheit degree-days), corresponding to a compressive strength on the order of 1.5 MPa (220 psi). Of the 18 lifts cast in the spring and used to support construction loads, 11 had a maturity similar to that of Lift 28 when loads were applied to them.

Lew and Fattal [3] conclude that concrete of this maturity and strength could resist the applied loads, although with a minimal safety margin, depending on the load effects from the hoisting system. After Lift 25 was placed, the static line for one cathead crane was moved to a new position, which significantly increased its load effect. As also noted in Ref 3, following this change in the hoisting system, Lift 28 was the first lift to be loaded after only one day of curing. A reason for this may be seen in Table 2. Weather information indi-

Date, April 1978	Day	Lift Placed	Average Temperature, °C	Rainfall, mm
17	Monday	25	+11	0
18	Tuesday		+12	16
19	Wednesday	26	+14	4
20	Thursday		+7	1
21	Friday		+5	3
22	Saturday		+7	T <sup>a</sup>
23	Sunday		+9	Т
24	Monday	27	+13	0
25	Tuesday		+11	5
26	Wednesday	28	+12	Т
27	Thursday	29	+12	0

TABLE 2-Concreting schedule and weather after Lift 25.

"T indicates trace amounts of precipitation, less than 0.5 mm.

cates that several days of rain may have disrupted the concreting operations during the last two work weeks of construction. The information in Table 2 shows that only Lift 26 was placed on a day with appreciable rainfall. Details of the monthly summary of the climatological data [13] show that the rainfall on that day occurred between midnight and 3 A.M. before the workday, and after 1 P.M., when concreting operations were likely to be winding down. These rain delays, in conjunction with the weekend of 22-23 April 1978 appear to have delayed the collapse until the placement of concrete in Lift 29. Lew and Fattal [3] estimate that a concrete strength of about 7 MPa (1000 psi) with a maturity on the order of 50 Celsius degree-days (90 Fahrenheit degree-days) was necessary to prevent initiation of failure for the hoisting loads resulting from the relocated static line. The maturity of the concrete supporting placement of Lifts 26, 27, and 28 was in the range of 45 to 94 Celsius degree-days (80 to 170 Fahrenheit degree-days). Also, the in situ maturity would tend to be somewhat higher because of the temperature increase resulting from the cement hydration process.

#### Conclusions

Although a precise determination of the margin of structural safety at any time during construction requires information on loadings, material strength, and structural configuration, much useful information can be obtained from an index of strength behavior, such as the maturity of the concrete.

In this study the Willow Island cooling tower collapse has been reviewed from a maturity viewpoint; that is, the maturity of the concrete in the tower shell provides an index to its material strength. This is particularly important in view of the scaffold system, which relied on the partially completed structure to resist construction loads. The pace of construction in the spring routinely caused a lift of concrete to be loaded by the scaffold system after only one day of curing. A change in configuration of the hoisting system following the placement of Lift 25 significantly increased stresses in the lift supporting new construction. Because of apparent rain delays and an intervening weekend, Lift 28 was the first lift to be loaded after only one day's curing for the new arrangement of the hoisting system. It appears likely that the collapse might have happened sooner if these various delays had not occurred.

#### Acknowledgments

This paper is based, in part, on the second author's problem report (prepared in partial fulfillment of the requirements for the M.S. degree in civil engineering) [5]. The authors would like to acknowledge the cooperation of P. Ianelli of the Parkersburg, West Virginia, office of the National Weather Service for assistance in obtaining the weather data used herein.

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# Summary

The first paper in this volume, that by Aitcin et al, discusses how curing conditions in the Arctic affect the strength gain for concrete specimens. Basically, two sets of experiments were carried out to simulate curing conditions for concrete caisson construction in the Arctic. One set of specimens was cured at  $0^{\circ}$ C, after 9 h of curing at 39°C. The second set was cured in a standard manner for comparison purposes. It was established that the concrete cured at  $0^{\circ}$ C achieved its 28-day compressive strength at the 56-day age. However, its modulus of elasticity took longer to achieve the equivalent 28-day design value based on standard curing conditions.

The paper by Berner, Gerwick, and Polivka discusses effects of cryogenic temperatures (up to  $-196^{\circ}$ C) on the behavior of high-strength lightweight concrete made with expanded shale aggregates. The key parameters investigated were the compressive and tensile strengths, modulus of elasticity, moisture content, and cyclic loading. The mechanical properties generally increased at low temperatures, with higher gains for specimens with increased moisture content. The cyclic loading induced relatively minor damage. The authors conclude that high-strength lightweight concrete should perform well, even at the cryogenic temperatures encountered in offshore containment vessels.

Carette and Malhotra provide results of a study undertaken to evaluate the performance of limestone and dolostone aggregate concretes subjected to temperatures in the range of 75 to  $600^{\circ}$ C. The test results show that the dolostone aggregate concrete is unstable under a sustained temperature exposure of 150°C. The limestone concrete was unaffected under similar exposures. It was also found that, as the temperatures increased beyond 150°C, the strength decreased with increasing temperatures and increasing exposure time. The pulse velocity and resonance frequency measurements were taken for monitoring compressive strength loss.

The next paper is by Gaynor, Meininger, and Khan. Their research shows that the increased water required for concretes produced at  $35^{\circ}C$  ( $95^{\circ}F$ ), and the subsequent strength loss, can be compensated for by a very modest amount of additional cement. It was determined that an increase in concrete temperature from 18 to  $35^{\circ}C$  (65 to  $95^{\circ}F$ ) required an average increase of about 4.7 kg (8 lb) of cement to maintain the specified strength levels. On the other hand, an increase in delivery time from 20 to 90 min required an additional 13.6 kg (23 lb) of cement.

The paper by *Mittelacher* also discusses effects of hot weather conditions on the strength of concrete. Data were collected from seven different projects to study the effects of hot weather conditions on the 28-day compressive strength. In general, the test specimens were left exposed to ambient hot weather conditions during the initial curing periods. A statistical analysis was performed on these data. However, no significant correlation was found between the placing temperatures and the strengths of these set-retarded concretes.

The paper by *Naik* examines the validity of the Nurse-Saul maturity function for concrete cured under winter curing conditions. The author concludes that the Nurse-Saul function should not be used for maturity-strength relationships for winter curing conditions. He establishes that the Arrhenius function should be used instead. Data are presented showing maturitystrength relationships determined by both of these functions.

Nasser and Chakraborty present results of an investigation of the influence of temperature on the structural properties of concrete containing Class F fly ash and a superplasticizer. Results show that up to  $71^{\circ}$ C ( $160^{\circ}$ F), the strength and elasticity of sealed and mass concrete were not greatly affected. At higher temperatures, 121 to 232°C (250 to 450°F), the strength and elasticity of mass concrete decreased, while the unsealed concrete was not significantly affected. The superplasticizer used did not seem to influence the properties of hardened concrete containing fly ash and exposed to high temperatures.

The paper by *Owens* discusses the effects of temperature fluctuations on the permeability of fly ash concrete. The research shows that temperature fluctuations increase the permeability of concrete. However, under similar conditions the permeability of fly ash concrete was reduced.

Roy, White, and Nakagawa examine the behavior of slag cements in comparison with that of portland cements. The effects of elevated temperatures up to 250°C, on mortars and pastes are determined. Compressive strength, density, microstructure, permeability, and dimensional change were the properties studied. Compressive strengths up to 200 MPa and higher were found in some of the mortars. Some changes in pore structure were noted with elevated temperatures.

The last paper is by *Halvorsen and Farahmandnia*. It presents a case study, using the maturity method, of the Willow Island cooling tower collapse of April 1978. This paper has evaluated the concrete strength in the cooling tower at the time of collapse. It shows that the concrete maturity was low at a time when the tower was subjected to construction loads. It further concludes that the failure might have occurred sooner if several days of rainy weather had not apparently delayed the construction.

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