

Effects of elevated temperature exposure on heating characteristics, spalling, and residual properties of high performance concrete

L. T. Phan¹, J. R. Lawson² and F. L. Davis¹

(1) Structures Division, Building and Fire Research Laboratory, National Institute of Standards and Technology, MailStop 8611, Gaithersburg, MD, 20899-8611, U.S.A.

(2) Fire Safety Engineering Division, Building and Fire Research Laboratory, National Institute of Standards and Technology, Gaithersburg, MD, 20899-8641, U.S.A.

Paper received: February 18, 2000; Paper accepted: September 22, 2000

ABSTRACT

This paper describes results of NIST's experimental program that focuses on effects of elevated temperature exposure on residual mechanical properties of HPC. Residual mechanical properties were measured by heating the 102 x 204 mm cylinders to steady state thermal conditions at a target temperature, and loading them to failure after the specimens had cooled to room temperature. The test specimens were made of four HPC mixtures with water-to-cementitious material ratio (w/cm) ranging from 0.22 to 0.57, and room-temperature compressive strength at testing ranges from 51 MPa to 93 MPa. Two of the four HPC mixtures contained silica fume. The specimens were heated to a maximum core temperature of 450°C, at a heating rate of 5°C/min. Experimental results indicate that HPCs with higher original strength (lower w/cm) and with silica fume retain more residual strength after elevated temperature exposure than those with lower original strength (higher w/cm) and without silica fume. The differences in modulus of elasticity are less significant. However, the potential for explosive spalling increased in HPC specimens with lower w/cm and silica fume. An examination of the specimens' heating characteristics indicate that the HPC mixtures which experienced explosive spalling had a more restrictive process of capillary pore and chemically bound water loss than those which did not experience spalling.

RÉSUMÉ

Cet article décrit les résultats du programme expérimental au NIST sur les effets que des températures élevées ont sur les propriétés résiduelles du BHP. Les propriétés mécaniques résiduelles ont été mesurées par la méthode suivante : des cylindres (102 x 204 mm) sont d'abord chauffés jusqu'à l'équilibre à une température choisie, puis stabilisés à température ambiante ; enfin ils sont chargés en compression jusqu'à la rupture. Les échantillons étaient préparés à partir de quatre mélanges BHP avec des rapports eau/matériaux cimentaires (e/mc) entre 0.22 et 0.57. Les résistances à la compression de ces mélanges étaient entre 51 MPa et 93 MPa. Deux des mélanges contenaient de la fumée de silice. Les échantillons étaient chauffés jusqu'à une température interne de 450°C, à un taux de chauffage de 5°C/min. Les résultats expérimentaux indiquent que le BHP, avec une résistance à la compression initiale supérieure (e/c plus bas) et contenant de la fumée de silice, retient une résistance à la compression résiduelle plus élevée que les échantillons ayant une résistance à la compression plus basse (e/c plus élevé) et sans fumée de silice. Les différences entre les modules d'élasticité sont moins marquées. Par contre, la possibilité d'éclatement de surface est supérieure pour les échantillons avec le rapport e/mc plus bas et contenant de la fumée de silice. Un examen des caractéristiques thermiques des échantillons indique que les mélanges BHP qui explosent subissent une perte plus limitée d'eau capillaire et chimiquement absorbée en comparaison avec les échantillons sans éclatement de surface.

1. INTRODUCTION

High Performance Concrete (HPC) can be manufactured by most concrete plants due to the availability of a variety of additives such as silica fume, fly ash, blast furnace slag, and water reducing admixtures. HPC often offers significant economic, architectural, and structural advantages over conventional concrete, and is being used

more widely in structural applications, especially when high durability is desired.

It is well established that mechanical properties of concrete in general are adversely affected by thermal exposure [1 to 13]. However, the effects of thermal exposure on HPC's mechanical properties have been found to be more pronounced than the effects on conventional concrete. More importantly, when exposed to

Editorial Note

Dr. Long Phan is a RILEM Senior Member. He participates in the work of RILEM TC 129-MHT Test methods for Mechanical properties of concrete at High Temperature. The NIST is a RILEM Titular Member.

relatively rapid heating (above $1^{\circ}\text{C}/\text{min}$), HPC has been shown to be more prone to dramatic spalling failure. The observed spalling failures in laboratory conditions have been characterized from being progressive (continuous spalling of small scales on the specimen's surface when subjected to radiant heating) to explosive (sudden disintegration of the specimen accompanied by the release of a large amount of energy which projects the broken concrete fragments in all directions with high velocity). It has been theorized that the higher susceptibility of HPC to explosive spalling at high temperature is due, in part, to its lower permeability, which limits the ability of water vapor to escape from the pores. This results in a build-up of pore pressure within the concrete. As heating increases, the pore pressure also increases. This increase in vapor pressure continues until the internal stresses become so large as to result in sudden, explosive spalling. Spalling, however, has been observed on an inconsistent basis. Often, explosive spalling has occurred to only a few HPC specimens from a larger group of specimens that were subjected to identical testing conditions. This erratic behavior makes it difficult to predict with certainty under what conditions HPC will fail by explosive spalling.

NIST is conducting an ongoing research program which aims to develop a fundamental understanding of the effects of elevated temperature exposure on HPC performance and to quantify the influences of different concrete parameters on its spalling potential and engineering properties as a result of elevated temperature exposure. The research program includes examination of HPC properties at elevated temperatures (tested hot) as well as residual properties (tested at room temperature after heating and cooling). This paper presents results of the test series that deals with the residual mechanical properties of HPC. Results of the entire NIST research program will be reported in a later publication.

2. EXPERIMENTAL PROGRAM

2.1 Test variables and test method

Effects of three different w/cm ratios (0.22, 0.33, and 0.57), *i.e.* compressive strengths, and the inclusion of silica fume (0 and 10% as cement replacement) on residual properties and spalling characteristics of HPC at elevated temperature are examined in this paper.

A schematic showing the residual property test method is shown in Fig. 1. All tests are performed under the *steady-state* temperature test condition. Under this test condition, the specimen is heated without loading to a target core temperature T , selected in this test program to be 100°C , 200°C , 300°C , or 450°C , using a constant furnace heating rate. The ambient temperature is then held constant for a period of time t_1 until it is determined that the *steady-state* temperature in the specimen has been reached. Once that is attained, the specimen is allowed to cool to room temperature by natural cooling. The specimen is then loaded to failure at room temperature

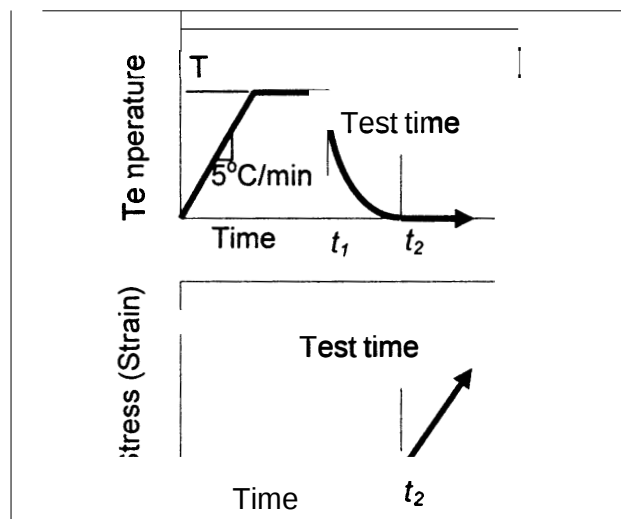


Fig. 1 - Residual property test method.

within 2 hours of the concrete core attaining room temperature level. The loading of the specimen follows the deformation control technique with a constant deformation rate of about $0.25\text{ mm}/\text{min}$. The ambient heating rate selected for this test program is $5^{\circ}\text{C}/\text{min}$, and the total temperature exposure time to attain steady-state temperature condition is 5 hr:30min. The selection of this exposure time is discussed in detail in the Heating Characteristics section. Steady-state temperature condition is defined as the temperature state when one of the following two conditions is attained:

- (1) the rate of temperature rise at the core of the specimen is less than $5^{\circ}\text{C}/\text{hr}$, or
- (2) the temperature at the core of the specimen reaches within $\pm 10^{\circ}\text{C}$ of the target temperature.

2.2 Test specimen

All specimens were made using Type I Portland cement, crushed limestone aggregate (13 mm nominal maximum size) with a fineness modulus (FM) of 5.40, natural sand fine aggregate with an FM of 2.85, silica fume, and a high range water-reducing admixture (HRWRA) based on a sulfonated naphthalene. The silica fume is in the form of a slurry with a density of $1.42\text{ g}/\text{cm}^3$ and a 54% silica fume concentration (by mass). Table 1 summarizes properties of the aggregates and silica fumes used.

Properties	Coarse Aggregate	Fine Aggregate	Silica Slurry
Density	1520 kg/m ³	1456 kg/m ³	1.418 g/cm ³
Fineness Modulus	5.40	2.85	-
Absorption (%)	-	0.59	-
Specific Gravity	2.6	2.63	1.35
Solids Content (% by mass)	-	-	54

Table 2 – Concrete mixture proportions

Materials	Mixture I (w/cm = 0.22)	Mixture II (w/cm = 0.33)	Mixture III (w/cm = 0.33)	Mixture IV (w/cm = 0.57)
Cement (kg/m ³)	595.9	595.9	661.6	376.4
Water (kg/m ³)	133.0	198.6	198.6	213.0
Coarse Aggregate (kg/m ³)	845.8	845.8	845.8	853.8
Fine Aggregate (SSD) (kg/m ³)	733.6	733.6	733.6	868.2
Silica Fume (kg/m ³)	65.7	65.7	0	0
Sulfonated naphthalene HRWR (ml)	400	354	154	0

Table 3 – Properties of fresh and hardened concretes

Properties	Mixture I (w/cm = 0.22)	Mixture II (w/cm = 0.33)	Mixture III (w/cm = 0.33)	Mixture IV (w/cm = 0.57)
Fresh Concrete				
- Slump (cm)	23.6	23.1	3.3	7.6
- Air Content (%)	3.2	2.75	2.0	2.5
Hardened Concrete				
- Initial Moisture Content (%)	5.03	6.07	6.26	7.33
- Compressive Strength (MPa)				
28-day	75.3	66.0	53.2	40.6
58-day	86.7	79.5	58.9	41.9
400-day	92.5	87.9	75.5	50.6
- Young's Modulus E* (GPa)				
58-day	34.4	37.2	36.7	34.4
400-day	47.2	43.7	44.1	36.7

* Dynamic Young's modulus determined using ASTM C215 – 91.

The specimens were made of four concrete mixtures, designated mixtures I to IV. Mixture I had the lowest w/cm of 0.22 and contained 10 % of silica fume as cement replacement. Mixtures II and III had the same w/cm of 0.33, and were designed to have similar strength but differ by the inclusion of silica fume (mixture II contained 10% of silica fume, while mixture III contained no silica fume). Mixture IV had the highest w/cm ratio of 0.57 and contained no silica fume. The mixture proportions are shown in Table 2. Sulfonated naphthalene based high range water-reducing admixture (HRWRA) was used in mixtures I, II, and III to improve the workability of the concretes.

Properties of fresh and hardened concretes are summarized in Table 3. Concrete room-temperature strength and modulus of elasticity were measured periodically starting from 28 days after casting. The earliest specimens tested at elevated temperature were more than 200 days after casting, at which time the increases in strength and modulus of all specimens have stabilized. Initial moisture contents were obtained by drying small concrete samples (400-day samples) at 105°C until the difference in mass losses between successive measurements is negligible (<0.1%). Dynamic modulus of elasticity was calculated from the resonant longitudinal frequency obtained from the Impact Resonant Method in accordance with ASTM C215-91.

All test specimens were 102 by 204-mm cylinders. The specimens were stripped of molds one day after casting and cured under water at room temperature (nomi-

nally 23°C) until close to test time. About one week before test time, the specimens were removed from the curing tank and both ends of each specimen were ground to meet ASTM C469 specifications for perpendicularity (ends are within $\pm 0.5''$ relative to specimen's longitudinal axis) and planeness (all points on each end are within 0.050 mm). The ground specimens were then placed under water again until 1 hour before testing.

Two specimens from each concrete mixture were each instrumented with three thermocouples to develop the internal temperature profiles on the cross section of the test specimens. One thermocouple was located at the center of the specimen (51 mm from side surface and 102 mm from ends). One was located midway between the center and the concrete surface (25 mm from side surface and 102 mm from ends). The third thermocouple was attached to the concrete surface at mid-

height (102 mm from ends). The thermocouples were 20-gage (0.8 mm) type K solid wire. The thermocouples' maximum temperature range is 980°C with a specified limit of error of 2.2°C or 0.75% for temperatures above 0°C. To place the two thermocouples that are inside the specimen, two 6.4 mm holes were drilled, within three days of test time, from one end to half the length of the specimen (102 mm from end). The thermocouples were inserted into the holes, which were then filled with cement mortar. The surface thermocouple was attached to the concrete surface by high temperature wire. Fig. 2 shows the specimen's dimensions and thermocouples' locations.

2.3 Temperature control

The instrumented specimens were heated using a heating rate of 5°C/min and temperature inside the concrete were recorded to provide information for temperature control, *i.e.* the temperature exposure time necessary to achieve steady-state thermal conditions. The heating rate of 5°C/min refers to the rate of temperature rise inside the furnace, and not the temperature rise inside the concrete specimen.

Figs. 3 and 4 show the temperature development and surface-to-core temperature gradient in a mixture I specimen due to heating. As shown in Fig. 3, after slightly more than 5 hours of heating at an ambient temperature

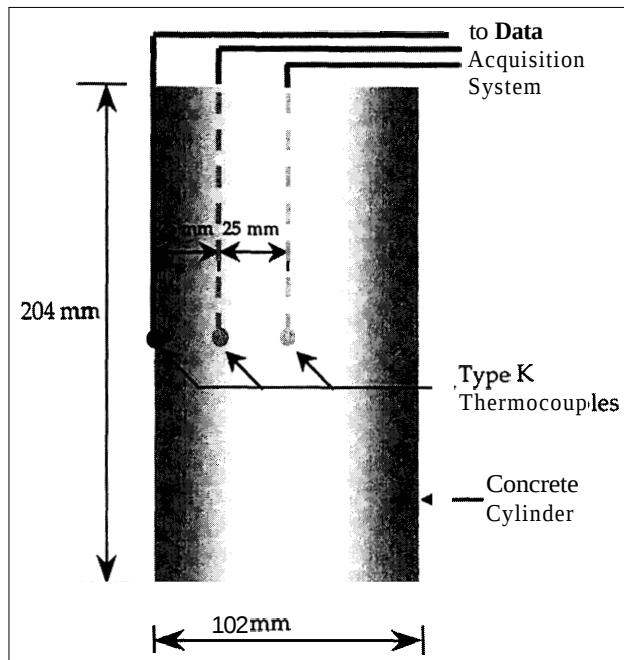


Fig. 2 - Specimen dimensions and instrumentation scheme.

of 500°C, the specimen core attained the temperature level of 450°C, with a surface-to-core thermal gradient of about -6°C (the concrete core is hotter than the concrete surface at this point). This satisfies the criteria for the steady-state thermal condition as outlined in the *Test Method* section. For cooling, experimental results indicated that it would take approximately 20 hours for the specimen core to cool to room temperature. Thus, referring to Fig. 1, a heating time t_1 of 5 hr:30 min & 15 min was selected as the standard heating time, and a test time t_2 of between $t_1 + 20$ hr and $t_1 + 22$ hr was selected as the standard test time in this test program. Also, note that the concrete core temperature is about 50°C lower than the ambient furnace temperature at the end of the heating period. Thus, in order to achieve the steady-state temperature condition at the selected specimen core temperatures of 100°C, 200°C, 300°C, and 450°C, it is necessary to heat the furnace to ambient temperatures 50°C higher

than the target test temperatures, i.e. 150°C, 250°C, 350°C, and 500°C.

3. EXPERIMENTAL RESULTS

Table 4 summarizes the measurements of heat-induced mass loss and changes in Young's modulus of elasticity and compressive strength of the test specimens. Measurement accuracy is expressed in terms of standard deviation (SD) and coefficients of variation (CV), which are also listed in Table 4. The convention used in naming the test specimens is as follows:

Test Method - Concrete Mixture -
(Residual Strength) (I to IV)

Target Core Temperature - Specimen Number
(23°C to 450°C) (1, 2, 3...)

3.1 Heating characteristics

Fig. 3 also provides information on the heating characteristics of the specimens, i.e. how elevated temperature exposure affects temperature development and moisture (evaporated capillary pore water and chemically bound water) movement inside the concrete cylinders. In this figure, the thick solid line represents the ambient temperature inside the furnace. The thin solid line is the temperature measured on the concrete surface. The broken dashed line is the temperature at a point inside the concrete, 25 mm from the concrete surface (mid-depth). The dotted line is the temperature at the concrete core, 51 mm from the concrete surface (see Fig. 2). Fig. 4 shows the thermal gradient between the specimen's surface and core during the heating exposure.

The first two vertical dashed lines from the left in Fig. 4 mark the perturbations in the rates of temperature rise between the concrete surface and core, at 1 hr: 15 min and 1 hr: 55 min into heating. These coincide with concrete core temperatures of slightly above 100°C and approximately 205°C, as marked by the same two vertical dashed lines on Fig. 3. The perturbations in rates of temperature rise between the concrete surface and core are believed to be due to the releases of capillary pore and chemically bound water beginning at these two temperatures. At slightly above 100°C, capillary pore water in mixture I concrete begins to evaporate. A moisture front is driven by the heat toward the core of the specimen, causing a decrease in the rate of temperature rise at the specimen core and thus an increase in the thermal gradient between the specimen's surface and core. Beginning at approximately 205°C, significant chemically-bound water in mixture I concrete is released. This caused a similar decrease in the rate of temperature rise at the core, as marked by the

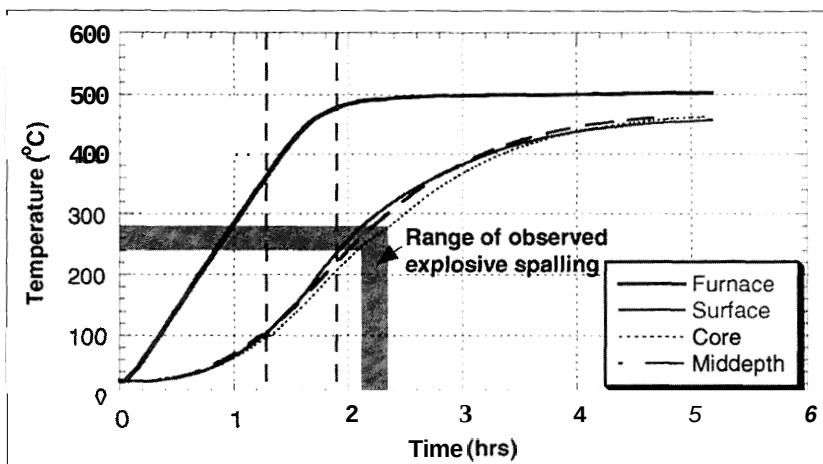


Fig. 3 - Temperature development inside mixture I cylinder.

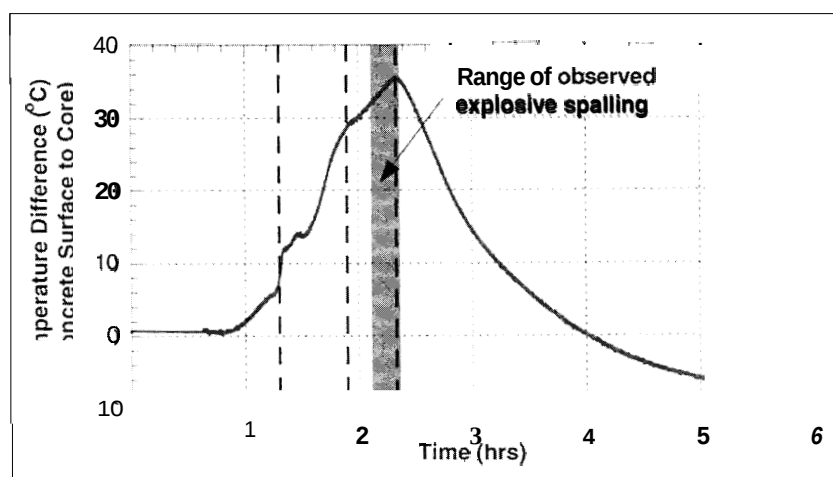


Fig. 4 - Thermal gradient between surface and core of mixture I cylinder.

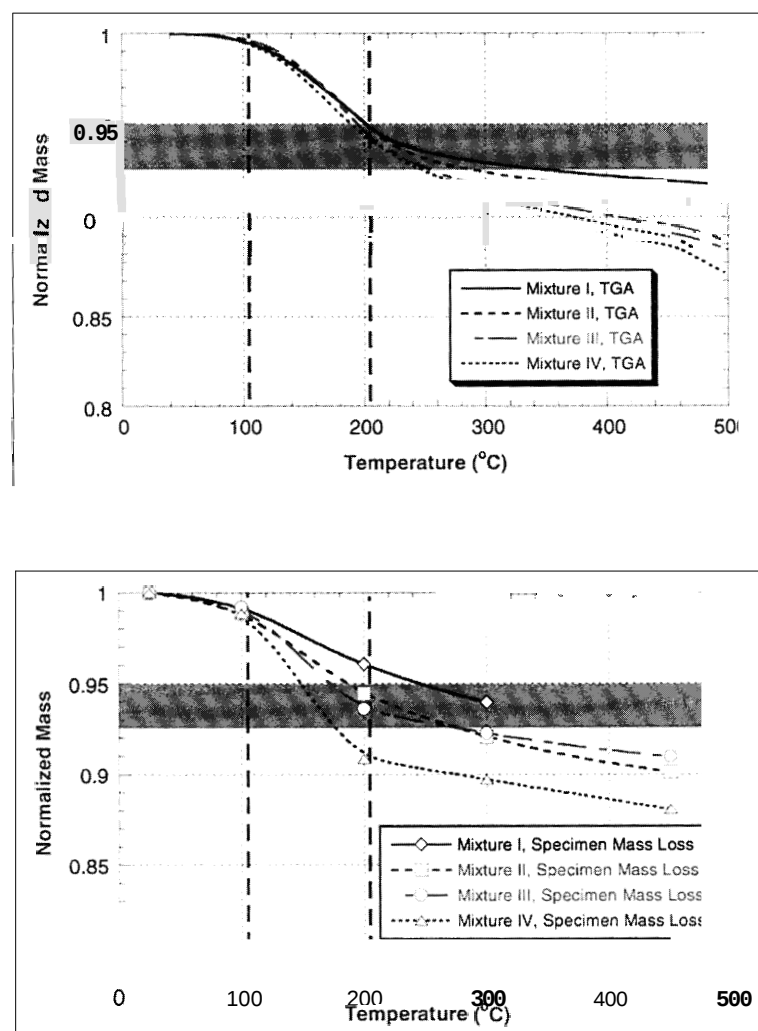


Fig. 6 - Mass losses from heating of full-size cylinders.

second dashed line in Figs. 3 and 4.

Thermal gradient between the specimen surface and core reaches a maximum of 36°C after 2 hr: 20 min of heating, at a corresponding core temperature of 270°C. After this point, the rate of temperature rise on the concrete surface begins to decrease faster than that of the core, causing the thermal gradient to decrease as shown

in Fig. 4. This trend continues until a true steady-state thermal condition develops after 4 hr of heating, when the surface-to-core thermal gradient is reduced to zero. After 5 hr:15 min of heating, the specimen core reaches the target temperature of 450°C and is about 6°C higher than the concrete surface.

Heat-induced mass losses for all four concrete mixtures are shown in Figs. 5 and 6. Fig. 5 shows mass losses obtained from thermogravimetric analysis (TGA) of small samples (approximately 100 mg each) taken from the four concrete mixtures [14].

Fig. 6 shows mass losses obtained from heating full-size (102 × 204 mm) cylinders to target temperatures of 100°C, 200°C, 300°C, and 450°C at 5°C/min. The two vertical dashed lines in each of these figures mark the changes in rates of mass loss. The TGA results show that, beginning at slightly above 100°C (first vertical dashed line in Fig. 5), all four mixtures sustain similar rate and amount of mass loss. This coincides with the change in the rate of temperature rise between surface and core of mixture I specimen, *i.e.* the beginning of the capillary pore and chemically bound water removal process discussed above (see Fig. 3). A slower rate of heat-induced mass loss begins at about 215°C (second vertical dashed line in Fig. 5) for all four concrete mixtures. While the mass loss rates were not significantly different for the four mixtures, the amounts of mass loss varied. Mixtures III and IV ($w/cm = 0.33$ and 0.57 , respectively), which contained no silica fume, sustained similar but larger amounts of heat-induced mass loss than mixtures I and II ($w/cm = 0.22$ and 0.33), which contained silica fume. As discussed above, the mass losses at this stage are due primarily to the release and evaporation of chemically bound water in the concrete samples. Fig. 6 shows mean mass losses in full-size (102 × 204 mm) cylinders, heated in accordance with the temperature control regime selected for this test program, *i.e.* a heating rate of 5°C/min and 5 hr:15 min exposure time. At each target temperature level, mass loss was measured for a group of at least three specimens and the results, along with the CV are listed in Table 4. The results show that m_{aw}

losses in mixture III and IV specimens follow the same two stages that begin at slightly above 100°C and 200°C as observed in the TGA measurements, with the mixture IV specimen sustaining the highest amount of mass loss. However, the changes in rate of mass loss for mixtures I and II at above 200°C are less apparent, with the mixture I specimen sustaining no change in rate of mass loss up to

Table 4 - Summary of test results

	Test Name	Mass Loss (%)	SD/CV of Mass Loss (%)	E Before Heating (Pa)	E After Heating (Pa)	Residual E (%)	SD/CV of Residual I (%)	Test Strength (MPa)	Residual Strength (%)	SD/CV of Residual Strength (%)	Failure Mode
Mixture I	RS-I-25-1	0.00		4.73E+10		100.0		90.56	100.0	3.6/3.6	Spalling
	RS-I-25-2	0.00		4.71E+10		100.0		90.60	100.0		
	RS-I-25-3	0.00		4.71E+10		100.0		96.40	100.0		
	RS-I-100-1	1.02	.14/15.8	4.60E+10	3.47E+10	75.5	5.8/7.0	75.64	81.8	5.5/6.4	
	RS-I-100-2	0.75		4.41E+10	3.76E+10	85.2		80.34	86.8		
	RS-I-100-3	0.84		4.24E+10	3.63E+10	85.7		85.92	92.9		
	RS-I-200-1	4.38	.41/10.5	4.46E+10	2.98E+10	66.7	3.5/5.0	93.29	100.8	3.6/3.8	
	RS-I-200-2	3.57		4.68E+10	3.45E+10	73.6		87.84	94.9		
	RS-I-200-3	3.85		4.67E+10	3.34E+10	71.4		87.08	94.1		
	RS-I-300-1	6.19	.11/1.7	4.61E+10	2.02E+10	43.8	0.6/1.4	82.85	89.5	3.8/4.4	
	RS-I-300-2			4.70E+10							
	RS-I-300-3	6.02		4.54E+10	1.97E+10	43.3					
	RS-I-300-4	6.07		4.71E+10	2.00E+10	42.4		76.41	82.6		
	RS-I-300-5	5.94		4.72E+10	2.05E+10	43.5		82.19	88.8		
	RS-I-450-1			4.81E+10							Spalling
	RS-I-450-2			4.65E+10							Spalling
	RS-I-450-3			4.59E+10							Spalling
Mixture II	RS-II-25-1	0.00		4.32E+10		100.0		88.87	100.0	0.9/0.9	Spalling
	RS-II-25-2	0.00		4.39E+10		100.0		87.66	100.0		
	RS-II-25-3	0.00		4.40E+10		100.0		87.31	100.0		
	RS-II-100-1	1.22	.07/5.8	4.25E+10	3.94E+10	92.6	1.6/1.7	78.53	89.3	4.6/5.3	
	RS-II-100-2	1.13		4.17E+10	3.92E+10	94.1		79.11	90.0		
	RS-II-100-3	1.09		4.36E+10	3.97E+10	91.0		71.78	81.6		
	RS-II-200-1	6.03	.48/8.6	4.34E+10	2.86E+10	65.8	3.2/4.6	67.75	77.0	2.0/2.6	
	RS-II-200-2	5.08		4.29E+10	3.10E+10	72.1		69.84	79.4		
	RS-II-200-3	5.55		4.37E+10	2.98E+10	68.1		71.32	81.1		
	RS-II-300-1	8.09	.15/1.8	4.40E+10	1.88E+10	42.8	2.0/4.5	58.84	66.9	2.6/3.9	
	RS-II-300-2	7.80		4.31E+10	1.98E+10	45.9		56.62	64.4		
	RS-II-300-3	7.91		4.30E+10	2.00E+10	46.6		61.22	69.6		
	RS-II-300-4										
	RS-II-450-1	9.16	1.1/ 11.4	4.36E+10	1.13E+10	26.0	0.5/2.1	41.32	47.0	1.5/3.3	
	RS-II-450-2	9.30		4.50E+10	1.12E+10	25.0		43.78	49.8		
	RS-II-450-3	11.17		4.36E+10	1.10E+10	25.2		41.40	47.1		
Mixture III	RS-III-25-1	0.00		4.41E+10		100.0		75.43	100.0	1.5/1.5	
	RS-III-25-2	0.00		4.34E+10		100.0		76.54	100.0		
	RS-III-25-3	0.00		4.48E+10		100.0		74.23	100.0		
	RS-III-100-1	0.83	.12/13.9	4.20E+10	3.85E+10	91.8	3.9/4.2	58.12	77.1	3.4/4.5	
	RS-III-100-2	0.69		4.13E+10	4.06E+10	98.4		55.94	74.2		
	RS-III-100-3	0.88		4.05E+10	3.88E+10	95.8		59.08	78.4		
	RS-III-100-4	0.97		4.25E+10	3.80E+10	89.4		53.31	70.7		
	RS-III-200-1	6.73	.29/4.5	4.33E+10	2.80E+10	64.6	4.0/5.7	59.58	79.0	4.7/6.4	
	RS-III-200-2	6.18		4.25E+10	3.05E+10	71.8		52.47	69.6		
	RS-III-200-3	6.30		4.19E+10	2.98E+10	71.0		56.73	75.2		
	RS-III-300-1	8.09	.30/3.9	4.24E+10	1.94E+10	45.7	1.4/2.9	55.54	73.7	2.7/3.6	
	RS-III-300-2	7.54		4.31E+10	2.08E+10	48.3		57.52	76.3		
	RS-III-300-3	7.61		4.36E+10	2.08E+10	47.7		53.50	70.9		
	RS-III-450-1	9.31	.45/4.9	4.33E+10	1.16E+10	26.9	0.6/2.2	41.75	55.4	3.4/6.6	
	RS-III-450-2	8.54		4.50E+10	1.17E+10	25.9		36.60	48.5		
	RS-III-450-3	9.31		4.33E+10	1.16E+10	26.9		38.72	51.3		
Mixture IV	RS-IV-25-1	0.00		3.67E+10		100.0		51.91	100.0	3.0/3.0	
	RS-IV-25-2	0.00		3.67E+10		100.0		51.03	100.0		
	RS-IV-25-3	0.00		3.67E+10		100.0		48.97	100.0		
	RS-IV-100-1	1.33	.24/21.1	3.72E+10	3.05E+10	82.2	0.7/0.8	35.21	69.5	1.6/2.3	
	RS-IV-100-2	0.88		3.70E+10	3.10E+10	83.6		35.16	69.4		
	RS-IV-100-4	1.27		3.75E+10	3.11E+10	82.9		36.58	72.2		
	RS-IV-200-1	9.53	.34/3.7	3.65E+10	1.85E+10	50.7	6.6/11.4	38.82	76.7	2.6/3.6	
	RS-IV-200-2	8.96		3.65E+10	2.32E+10	63.6		36.45	72.0		
	RS-IV-200-3	8.94		3.73E+10	2.22E+10	59.5		36.58	72.2		
	RS-IV-300-1	9.59	.92/9.0	3.66E+10	1.27E+10	34.8	3.6/9.3	34.91	68.9	2.1/3.2	
	RS-IV-300-2	9.93		3.70E+10	1.53E+10	41.2		33.15	65.5		
	RS-IV-300-3	11.33		3.67E+10	1.50E+10	41.0		32.96	65.1		
	RS-IV-450-1	11.96	.35/2.9	3.67E+10	7.96E+09	21.7	0.3/1.5	27.06	53.4	2.8/5.4	
	RS-IV-450-2	12.30		3.63E+10	7.84E+09	21.6		25.46	50.3		
	RS-IV-450-3	11.61		3.74E+10	8.29E+09	22.2		24.29	48.0		

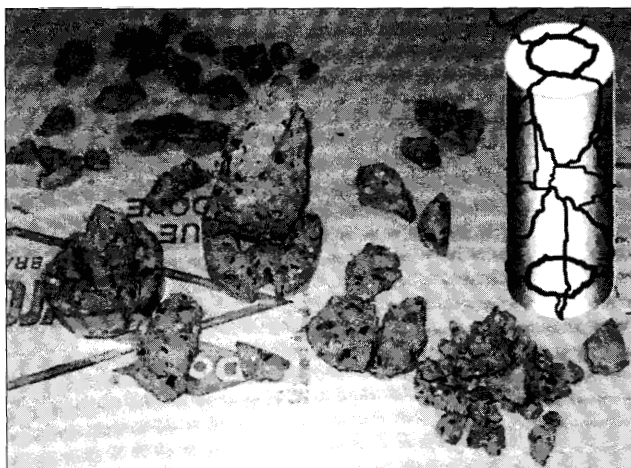


Fig. 7 - Remnants of an exploded cylinder and rendering of the fracture formation.

300°C. It should be noted that mass loss data for mixture I specimens at 450°C are not available due to explosive spalling of all three specimens while being heated to that temperature level. More detailed discussion concerning the explosive spalling of this group of specimens is given in the next section.

Recall that the initial moisture contents measured for the four concrete mixtures in this test program ranged between 5.0% for mixture I and 7.3% for mixture IV (see Table 3). Initial moisture contents represent the amount of capillary pore water in the concrete matrix and were obtained by drying small concrete samples (400-day samples) at 105°C until the difference in mass losses between successive measurements is negligible ($\leq 0.1\%$). The total losses of capillary pore water in all four concrete mixtures are represented by the horizontal band in Figs. 5 and 6 (at normalized masses of 0.93 and 0.95). The TGA results, shown in Fig. 5, indicate that all four mixtures sustained significant loss of capillary pore and chemically bound water at about 215°C (marked by the second vertical dashed line). However, the results of heating full-size specimens, shown in Fig. 6, indicate that while specimens of mixtures II, III, and IV appear to lose most of this water at 215°C, the water in mixture I specimen was not completely liberated at this temperature (only 4%). Similarly, but to a lesser extent, the mixture II specimens also experienced a slightly more restrictive process of heat-induced water loss (5.5%) compared with the TGA result. These differences in mass loss between the TGA and full specimen heating indicate that, while mixture III and IV cylinders will have little problem losing water at high temperatures, the silica fume containing mixtures I and II cylinders will have a more restrictive water loss process and could thus develop significant internal pressures (leading to spalling).

3.2 Spalling characteristics

As listed in Table 4, explosive spalling occurred in five specimens, four of mixture I concrete and one of mixture II, in this test series. Both mixtures I and II

contained 10 percent of silica fume as cement replacement. Explosive spalling is characterized by the sudden disintegration of the test specimens into fine fragments during heating. This disintegration is accompanied by a sharp loud bang and the release of a sufficient amount of energy which projects the small concrete fragments at high velocity in all directions. Reconstruction of the exploded specimens shows that the largest remaining piece in all cases is the concrete core, which measured approximately 70 mm at maximum width and 120 mm at maximum length. This core is surrounded by an approximately 20-mm thick concrete outer shell along the length of the cylinder. The depth of approximately 20 mm appears to be the location of the primary fracture surface. Fig. 7 shows the fragments of an exploded specimen and a rendering of the fracture formation based on reconstruction using these fragments.

Of the four exploded mixture I specimens, one belongs to a group of five specimens being heated to a target temperature of 300°C (specimen RS-I-300-2). This specimen exploded at 2 hr: 5 min into the heating process, with the furnace temperature being maintained at 300°C and the specimen core temperature of 240°C. The other three exploded mixture I specimens are of the entire group of three specimens being heated to 450°C (specimens RS-I-450-1 to 3). These specimens exploded successively, beginning at a core temperature of 240°C and ending at 280°C. The exploded mixture II specimen (RS-II-300-4) belongs to a group of four mixture II specimens being heated to 300°C. This specimen exploded at a core temperature of 275°C. The temperature range in which explosive spalling occurred in mixture I specimens is superposed over the temperature profile and thermal gradient for mixture I concrete and represented by the shaded bands in Figs. 3 and 4. As can be seen in Fig. 4, the temperature range in which explosive spalling occurred coincides with the time when maximum thermal gradient between the specimen surface and core occurred. This suggests that, while internal pore pressure may be the primary cause for the explosive spalling of the specimens, as evidenced by the high velocity and force with which the concrete fragments were projected at failure, the buildup of thermally induced strain energy was also at a maximum at this time, and thus thermal stress might have a secondary role in this failure.

3.3 Residual mechanical properties

Compressive strengths of original (23°C) and heated specimens are listed in Table 4 and plotted with respect to the target temperatures they were exposed to in Fig. 8. Also listed in Table 4 are residual strengths of heated specimens, normalized with respect to the means of original compressive strengths, and the coefficients of variation (CV) and standard deviation (SD) for each group of specimens. As shown, the CV for compressive strength measurements varies from 0.9 to 6.6 percent. The maximum CV of 6.6 percent is within the accept-

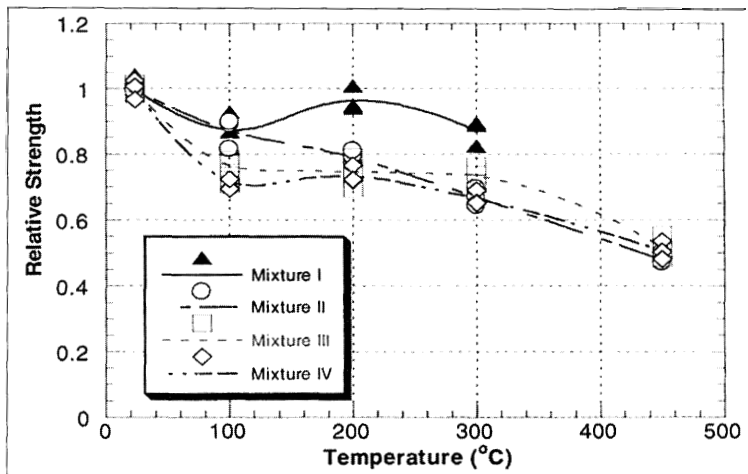


Fig. 8 - Residual compressive strength vs. temperature.

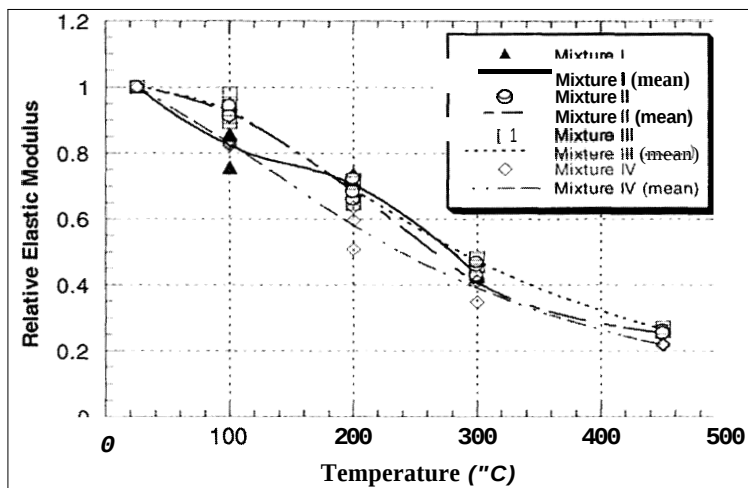


Fig. 9 - Residual dynamic Young's modulus of elasticity vs. temperature.

able range of 7.8 percent specified by ASTM C39 for groups of three or more specimens tested in laboratory conditions.

Plotted in Fig. 8 are both the individual test data (symbols) at each temperature level and the means (lines) of these test data. As shown in Fig. 8, compressive strengths of mixtures III and IV concretes, which had w/cm ratios of 0.33 and 0.57 respectively and contained no silica fume, varied similarly with increasing temperature. The strength reduction in these two mixtures can be characterized by an initial strength reduction of between 25 to 30 percent at 100°C. This is followed by a period of no significant change in compressive strength between 100°C to 300°C. Further reduction in compressive strength resumes at temperatures above 300°C. Exposure to 450°C caused a 50 percent compressive strength loss in mixture III and IV concretes.

The silica fume containing concretes, mixtures I ($w/cm = 0.22$) and II ($w/cm = 0.33$), had a similar strength reduction of between 10 to 15 percent at 100°C. At temperatures above 100°C, mixture II strength continued to drop - almost linearly - with increasing temperature, while mixture I concrete experienced a strength recovery at temperatures between 100°C and 200°C.

Between 200°C and 300°C, mixture I strength decreased at a similar rate as that of mixture II. However, mixture I concrete sustained only 10 to 20 percent strength loss at 300°C, while strength loss for mixture II almost doubled, at 30 to 35 percent, at this temperature. At 450°C, mixture II specimen sustained similar amount of strength loss, about 50 percent, as for mixtures III and IV. Compressive strength data for mixture I concrete at 450°C were not available due to explosive spalling of the entire group of specimens (RS-450-1-1 to 3) while being heated to this temperature.

These results indicate that, within the ranges of original compressive strengths (51 MPa to 93 MPa) and w/cm ratios (0.22 to 0.57) examined in this test program, concrete with higher original compressive strength, or lower w/cm ratio, experienced less strength loss due to high temperature exposure than concrete with lower original compressive strength. In concretes with similar w/cm ratio and strength (mixture II and 111), the presence of silica fume appears to result in lower strength loss up to the temperature of 200°C.

Dynamic Young's modulus of elasticity of each specimen measured before and after exposure to heating are listed in Table 4. The residual dynamic Young's modulus of elasticity, calculated for each specimen by normalizing the elastic modulus after heating by the value measured before heating, are also listed in Table 4. Also listed in Table 4 are the CV for the residual dynamic Young's modulus of elasticity, which ranges from 0.8% to 11.4%.

ASTM C215-91 does not specify criteria for an acceptable range of CV for the measurements of longitudinal response frequencies, i.e. dynamic Young's modulus, using the Impact Resonance Method.

Fig. 9 shows the variation of residual dynamic Young's modulus of elasticity of four concrete mixtures in this test program with increasing temperatures. The symbols in Fig. 9 represent individual test data, and the lines represent the means. As shown in this figure, dynamic Young's modulus of elasticity of the four mixtures of concrete in this test program decreased similarly with increasing temperature. Between room temperature (23°C) and 300°C, dynamic Young's modulus of elasticity for all mixtures decreased by more than 50 percent. Between 300°C to 450°C, the rate of modulus reduction decreased. Exposure to 450°C caused all four concrete mixtures to lose more than 70 percent of their initial dynamic Young's modulus of elasticity. Mixtures II and III, which have the same w/cm ratio of 0.33 and somewhat similar room-temperature compressive strengths (81 MPa and 72 MPa, respectively), display almost identical residual dynamic Young's modulus of elasticity. The reduction in modulus of mixtures II and III concretes are consistently less than (5 to 10 percent)

that of mixture IV, which has a w/cm of 0.57 and a room-temperature compressive strength of 51 MPa. The reduction in dynamic modulus of elasticity for mixture I concrete ($w/cm = 0.22$, 93 MPa) is less consistent within the range of temperatures examined and varied between those of mixture IV and mixtures II and III.

4. SUMMARY AND DISCUSSIONS

The results of one the NIST test series to study the effects of elevated temperature exposure, up to 450°C at the test specimen core, on heating characteristics, potential for explosive spalling, and residual mechanical properties of four HPC mixtures were reported. Other test series to study effects of heating exposure on performance of HPC at elevated temperatures will be reported in a later publication. The following summarizes experimental results and observations:

- The presence of silica fume in HPC mixtures with w/cm of 0.33 or less increases the potential for explosive spalling of 102 × 204 mm concrete cylinders. Explosive spalling occurred when the cylinders were being heated to target temperatures of 300°C and 450°C at an ambient heating rate of 5°C/min. The specimens' core temperatures measured when explosive spalling occurred were in the range of 240°C to 280°C, slightly beyond the temperatures at which much of the chemically bound water has already been released from the concrete matrix.

- Evidence of a more restrictive heat-induced mass loss process experienced by the exploded specimens, coupled with the sudden, dramatic disintegration of the test specimen into small fragments that project outward at high velocity, further support the hypothesis that internal pore pressure buildup is the primary cause for the explosive spalling mechanism. However, the fact that explosive spalling occurred at a time when the maximum thermal gradient also existed in the specimen implies that the buildup of strain energy due to thermal stress might also have a secondary role in the explosive spalling mechanism. Whether this secondary role is to delay or to contribute to the explosive spalling mechanism remains to be quantified (since the aggregates expand with increasing temperature while the cement paste contracts).

- Within the ranges of original compressive strengths (51 MPa to 93 MPa) and w/cm (0.22 to 0.57) examined in this test program, HPC with higher original compressive strength, or lower w/cm ratio, sustained lower strength loss due to high temperature exposure than those with lower original compressive strength. In HPC with similar w/cm and strength (mixture II and III), the presence of silica fume appears to result in lower strength loss up to the temperature of 200°C.

- Dynamic modulus of elasticity of the four HPC mixtures in this test program decreased similarly with increas-

ing temperature. Between room temperature (23°C) and 300°C, modulus of elasticity for all mixtures decreased by more than 50 percent. Between 300°C to 450°C, the rate of modulus reduction decreased. Exposure to 450°C caused all four concrete mixtures to lose more than 70 percent of their initial modulus of elasticity.

REFERENCES

- [1] Schneider, U., 'Properties of Materials at High Temperatures – Concrete', RILEM - Committee 44 - PHT, University of Kassel, Kassel, June 1985.
- [2] Phan, L. T., Carino, N. J., Duthinh, D. and Garboczi, E., 'NIST International Workshop on Fire Performance of High Strength Concrete', NIST, Gaithersburg, MD, February 13-14, 1997 – Proceedings, NIST SP 919, Gaithersburg, Maryland, September 1997.
- [3] Castillo, C. and Durrani, A. J., 'Effect of transient high temperature on high-strength concrete', *ACI Materials Journal* **87** (1) (1990) 47-53.
- [4] Hertz, K., 'Danish investigations on silica fume concretes at elevated temperatures', Proceedings of ACI 1991 Spring Convention, March 17-21.
- [5] Diederichs, U., Jumppanen, U. M. and Penttala, V., 'Behavior of high strength concrete at high temperatures', Helsinki University of Technology, Department of Structural Engineering, Report no. 92, 1989.
- [6] Hammer, T. A., 'High-strength concrete phase 3: Compressive strength and E-modulus at elevated temperatures', SP6 Fire Resistance, Report 6.1, SINTEF Structures and Concrete, February 1995.
- [7] Sullivan, P. J. E. and Shanshar, R., 'Performance of concrete at elevated temperatures (as measured by the reduction in compressive strength)', *Five Technology* **28** (3) (Aug 1992) 240-250.
- [8] Abrams, M. S., 'Compressive strength of concrete at temperatures to 1600°F', ACI SP 25, Temperature and Concrete, Detroit, Michigan, 1971.
- [9] Morita, T., Saito, H. and Kumagai, H., 'Residual mechanical properties of high strength concrete members exposed to high temperature - Part 1. Test on material properties', Summaries of Technical Papers of Annual Meeting, Architectural Institute of Japan, Niigata, August 1992.
- [10] Furumura, F., Abe, T. and Shinohara, Y., 'Mechanical properties of high strength concrete at high temperatures', Proceedings of the Fourth Weimar Workshop on High performance Concrete: Material Properties and Design, Weimar, Germany, October 4th and 5th, 1995, 237-254.
- [11] Felicetti, R., Gambarova, P. G., Rosati, G. P., Corsi, F. and Giannuzzi, G., 'Residual mechanical properties of HSC subjected to high-temperature cycles', Proceedings, 4th International Symposium on Utilization of High-Strength/High-Performance Concrete, Paris, France, 1996, 579-588.
- [12] Noumowe, A. N., Clastres, P., Debicki, G. and Costaz, J.-L., 'Thermal stresses and water vapor pressure of high performance concrete at high temperature', Proceedings, 4th International Symposium on Utilization of High-Strength/High-Performance Concrete, Paris, France, 1996.
- [13] Phan, L. T., 'Fire performance of High-Strength Concrete: A Report of the State-of-the-Art', NISTIR 5934, National Institute of Standards and Technology, Gaithersburg, Maryland, December 1996.
- [14] Bentz, D. P., 'Fibers, percolation, and spalling of high performance concrete', *ACI Materials Journal* **97** (3) (May-June 2000).