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Effects of Elevated Temperatures to High Strength Concrete with Nanosilica

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Abstract

High strength concrete has risen due to the demand for more complex shapes, sizes, forms, and strength of structures. High strength concrete with nanosilica exhibited improved strength both in tension and compression and improved durability with lower permeability outpacing normal strength concrete. It is falsely assumed that if a material has better mechanical properties at room temperature its performance is also better at higher temperature. The combined effects of calcium silicate hydrate (CSH) disintegration, contraction of cement paste, expansion of aggregates, and vapor pressure resulted to cracking and led to spalling. However, little to no significance is given to high strength concrete despite the irreversible effects of fire.

The research subjected cylindrical samples under peak temperature of 25°C, 100°C, 200°C, 450°C and 600°C for two hours with 0%, 2%, 3%, and 4% nanosilica by cement weight. High strength concrete has shown insignificant decrease in compressive strength and tensile strength due to CSH stability when subjected to temperatures less than 200°C. Residual compressive and tensile strength retained lowest under 450 °C and 600 °C. The reduction in strength is attributed to CSH disintegration, contraction of cement paste, and expansion of aggregate that led to shrinkage and weakening of the interfacial transition zone (ITZ). Significant increase in moisture loss is also observed when temperature reached 450°C. Spalling occurrences are small pop outs to none even after reaching 600°C. Under electron microscope, morphology of 3% nanosilica samples developed block-like medium with long continuous cracks while 0% samples are craterous with short discontinuous cracks. Microcracks are apparent beyond 200°C for all samples. The products of hydrated nanosilica provided fillers between gaps and voids but are weak enough that temperatures above 200°C splits the medium apart.

Keywords: high strength concrete; nanosilica; elevated temperatures

1. Introduction

High strength concrete (HSC) has gained more attention as structures are getting more and more complex in shapes, forms, strength and other specifications. Results from (Barbhuiya, [1–3] shown that HSC improved compressive strength, tensile strength and permeability improved with addition of nanosilica at certain percentages due to formation of secondary calcium silicate hydrates (CSH). But structures are seldomly designed under elevated temperatures due to its rare occurrence in its lifespan or worse little to no considerations [4]. It is a misconception that if concrete performs well at room temperature same behavior will happen at elevated temperatures [4]. If high strength concrete is subjected to elevated temperature, at 100°C free and adsorbed water evaporates. At 200°C, capillary water evaporates resulting to more adsorbed water loss. Furthermore at 450°C, aggregates will expand but cement paste will shrink due to loss of water in the matrix that will break the bond between cement paste and aggregates then CSH will start to disintegrate at 600 °C. With lower permeability due to secondary formation of CSH and relatively smaller particle size of nanosilica, concrete will exhibit higher water vapor pressure inside. If the vapor pressure exceeded the net tensile strength of the concrete, cracks will start to form and eventually lead to spalling of concrete layers [5]. Thus, exposing steel or tendons embedded to the matrix increasing risk in further damage and collapse. The combined effects of CSH disintegration, simultaneous contraction of cement paste and expansion of aggregate and increased vapor pressure can be detrimental to concrete.

2. Methodology

2.1. Material Proportions

Design mix was adapted from the [2], [6], [7] with water cement ratio maintained low to obtain low porosity of concrete. High water cement ratio leads to high workability but lower strength. Superplasticizer used is a polymer-based solution of chemical dispersant combined with other chemicals which is capable of reducing required water and achieve high strength requirement [8]. While retarder

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improved workability and setting time of fresh concrete. Nanosilica is a by-product of alkaline extraction and fast acid precipitation at room temperature by adding inorganic salts with surfactant to rice husk ash. Nanosilica used in the experiment is imported from China since the material is not readily available in the local market.

Table 1. Design Mix Proportions

Nanosilica Component	*0% (nS0)*	*2% (nS2)*	*3% (nS3)*	*4% (nS4)*
Slump (inches)	6-7	6-7	6-7	6-7
Water-cement Ratio by weight	0.28	0.28	0.28	0.28
Amount of Water (kg per cubic meter of concrete)	158	158	158	158
Cement (ASTM Type I) (kg/m^3)	564	564	564	564
Coarse Aggregate (kg/m^3)	1070	1070	1070	1070
Fine Aggregate (kg/m^3)	647	647	647	647
Superplasticizer, High-range Water Reducing Admixture (Type F) (L/m^3 of concrete)	11.6	16.86	20.43	24.64
Retarder (Type D) (L/m^3 of concrete)	1.12	1.12	1.12	1.12
Nanolisica (kg per kg of cement)	0	0.02	0.03	0.04

2.2. *Experiment Procedures*

Concrete mixing procedures were adapted from [9] with total of eight batches. Two batches were prepared for each variation of nanosilica, (20 samples each) total of 160 samples. Samples were cured by fully submerging in a water tank for 774 days. After air drying for 24 hours, samples were subjected to elevated temperatures peaked at 25°C, 100°C, 200°C, 450°C, and 600°C as seen below.

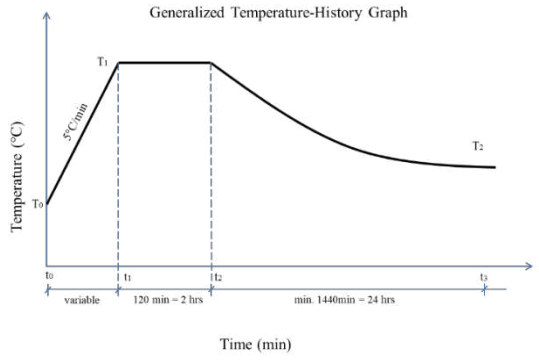


Figure 1. Generalized Temperature-History Graph

where:

T_0 – room temperature at test start
T_1 – maximum temperature set for respective samples
T_2 – steady state temperature after cooling down
t_0 - initial time
t_1 - total time to attain experimental peak temperature
t_2 - time of removal from the oven (t_2-t_1 = 2 hours)
t_3 - time for attaining thermal equilibrium with room temperature (t_3-t_2 =minimum of 24 hours)

3. Results and Discussion

3.1. *Compressive Strength*

Compressive strength of different nanosilica content has shown decrease for all samples. Remarkably at 3% nanosilica samples showed the least residual strength at 600°C due to continuous degradation of calcium silicate hydrates (CSH). Compressive strength gain is observed in all samples at 100°C. Similar to the observations of [10] that addition of nanosilica produced longer chain lengths of CSH and yielded to higher volume of solids in the matrix. On the other hand, significant decrease in strength is observed at 450°C for all samples with nanosilica due of simultaneous expansion of aggregates and shrinkage of CSH. This phenomenon lead cracks not just at void spaces but also to the ITZ causing the strength to decrease down to 23.53% of the original at the very least.

Temp (°C)	Strength in MPa				Residual Strength (%)			
	0%	2%	3%	4%	0%	2%	3%	4%
25	44.24	71.92	76.10	69.10	100.00	100.00	100.00	100.00
100	59.65	78.05	79.34	70.51	134.85	108.53	104.27	102.04
200	55.91	69.56	72.30	65.17	126.40	96.72	95.01	94.31
450	56.13	58.58	28.14	51.61	126.88	81.46	36.98	74.68
600	56.05	33.16	17.90	30.09	126.70	46.11	23.53	43.54

Table 2. Residual Compressive Strength

Rate of degradation can be observed on the slope of each line varying from 0.011, 0.0673, 0.1174 and 0.0668 for 0%, 2%, 3% and 4% respectively. See Figure (x-axis-temperature in °C, while y-axis is the residual strength in MPa). Slopes were translated from MPa per °C to kPa per °C for easier reference. Experiment and literatures have proven that concrete with nanosilica gained compressive strength, on the contrary, control samples (0% nanosilica) have the lowest rate of compressive strength degradation (0.0110) while 3% samples have the highest rate of compressive strength degradation (0.1174). Similarly, compressive strength of 0% nanosilica increases at 11.0 kPa per °C while the 3% decreases at 117.4 kPa per °C.

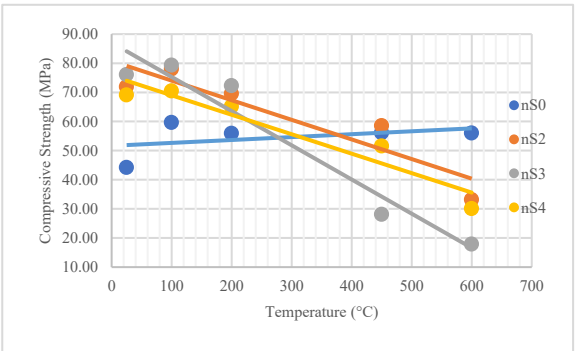


Figure 2. Compressive Strength vs Temperature at Varying Nanosilica

Label	Relation	R^2
0% (nS0)	$y=0.0110x+51.65$	0.1686
2% (nS2)	$y= -0.0673x+80.771$	0.8484
3% (nS3)	$y= -0.1174+87.04$	0.9431
4% (nS4)	$y=-0.0668x+75.662$	0.9123

Table 3. Temperature-Compressive Strength Relations

3.2. Modes of Failure

Modes of failure are based from [11]. Samples subjected to 100°C and 200 are both dominated by type 3 fractures. While under 450°C, types 4 and 5 both have 5 samples fractured each. And at 600°C, a combination of types 2 and 4 each have 4 samples fractured. Type 6 has the least number of samples with only 2. Followed by type 1 with 9 units. Types 2 and 5 have 14 and 16 fractured samples respectively. Eighteen (18) units formed the type 4 fracture in compression and 21 samples have shown type 3

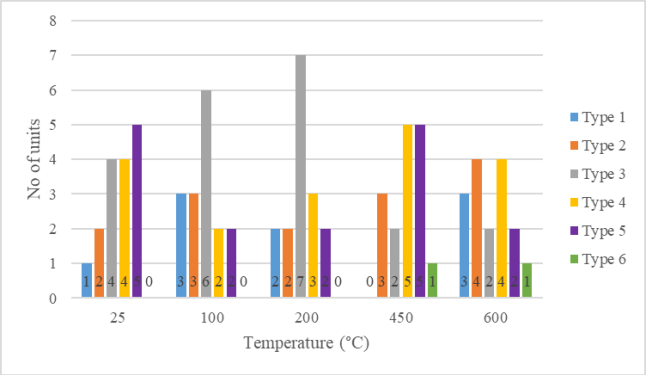


Figure 3. Modes of Failure in Compressive at varying Nanosilica

3.3. Tensile Strength

Tensile strength of all samples decreased regardless of nanosilica content. Rate of degradation can be observed on the slope of each line varying from 0.003, 0.0021, 0.0043 and 0.0026 for 0%, 2%, 3% and 4% samples respectively. Results from tensile strength tests confirm to the compressive tests, both showing decrease in strength as temperature increases.

Table 4. Residual Tensile Strength

Temp °C	Strength in MPa				Residual Strength (%)			
	0%	2%	3%	4%	0%	2%	3%	4%
25	3.46	3.82	4.00	4.03	100.00	100.00	100.00	100.00
100	3.34	3.26	4.10	3.54	96.61	85.31	102.52	87.93
200	3.92	3.62	3.34	2.91	113.26	94.80	83.60	72.22
450	2.22	3.38	2.31	3.12	64.12	88.44	57.77	77.38
600	1.98	2.22	1.72	2.10	57.17	58.11	43.14	52.05

Tensile strength of 0% samples decreased at 3.0 kPa per °C while samples with 3% nanosilica decreases at 4.3 kPa per °C. Also, transition is observed at 200°C wherein all samples converge at the 200°C. Notably, increase in strength is found random between 100°C and 200°C for all variations of nanosilica due to stress transfer in tension. Portions of concrete will not be able to transfer tensile stress if adjacent portions decomposed under elevated temperature unlike in compression, causing arbitrary results in increase in strength between 100°C and 200°C samples.

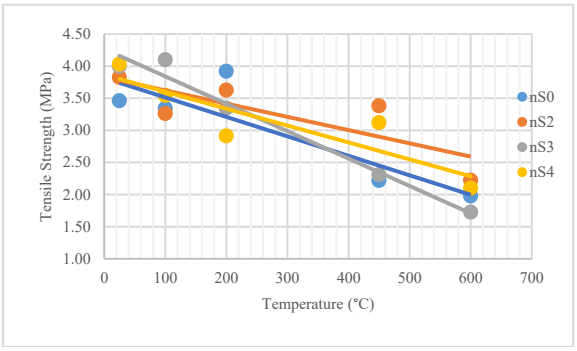


Figure 4. Tensile Strength vs Temperature at Varying Nanosilica

For T≥200°C, a gradual increase is observed for all samples including 0% samples. The contradicting results show that addition of nanosilica can impose a negative effect to concrete if subjected to elevated temperature.

Table 5. Temperature-Tensile Strength Relations

Label	Relation	R^2
0% (nS0)	$y=-0.0030x+3.8157$	0.7647
2% (nS2)	$y= -0.0021x+3.8288$	0.6478
3% (nS3)	y= −0.0043x+4.2668	0.9772
4% (nS4)	y=−0.0026x+3.8643	0.7769

3.4. Moisture loss and Spalling Severity

Moisture loss is determined by measuring the weights of cylinders before and after oven testing. Free and adsorbed water is less than 1% of the overall weight of the sample. While 3% nanosilica samples showed the largest moisture loss of 9.03% at 450°C. The 3% nanosilica samples showed optimum content with its increase in permeability among variety of samples ranging from 3% to 10% [2]. This large moisture loss is due to the loss of rich interlayer water trapped inside the denser concrete due to its improved permeability. At 450°C, aggregates start to expand and CSH will shrink at the same time, weakening the ITZ and creating microcracks. With the exposure of interlayer water, microcracks will start to interconnect with the capillaries creating pathways to relieve vapor pressure. The relief will lead to the large moisture loss and large increment compared to other samples.

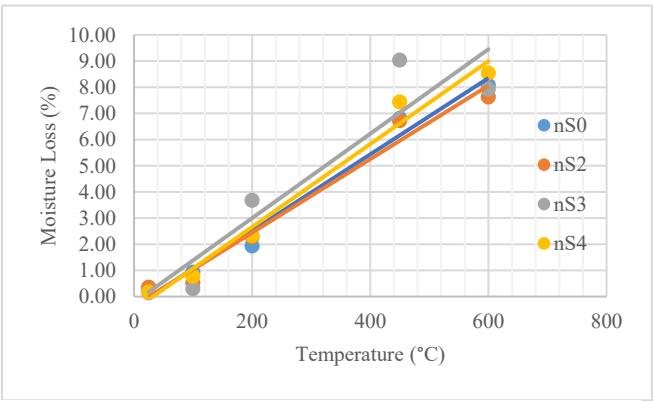


Figure 5. Moisture loss vs Temperature at varying Nanosilica

Thus, moisture loss can be a good indicator of loss of strength in controlled samples since concrete is dependent on water for its strength and if water is loss after hydration, it means that concrete has been disturbed.

Spalling severity is adapted from [12]. With most samples subjected to 25°C show little to no spalling with good original surface with only 89 samples are at level 1 spalling while 71 has reached level 2 spalling.

Table 6. Spalling Severity of Concrete

Severity Level	Description
1-Excellent	Good original surface, hard material
2- Very Good	Small chips or pop outs
	Surface spalling that exposed coarse aggregates
3- Good	Spalling of concrete with 1/3d to 1/2d in depth
4- Fair	10% to 15% of surface area is affected
5-Poor	Spalling is more than 15% area of beam or 30% area of slab
	Spalling depth > d
	d- depth

It is expected to increase the number of units with higher level of spalling due to crack formation caused by the higher temperature. From figure below, level 1 spalling (very good condition) reached 30 units at 25°C. The total units went down to 17, 12, 17 and 13 at 100°C, 200°C, 450°C, and 600°C respectively. See upper right legend for spalling severity level. Level 2 spalling (good condition) starts from 2 then increases to 15, 20, 15 and 19 at 25°C ,100°C, 200°C, 450°C, and 600°C respectively. As temperature increases, level 1 spalling decreases and level 2 spalling increases. As temperature increases, heat penetrates from surface to the core, water will escape as its natural reaction to heat and if the net vapor pressure exceeds the tensile capacity of concrete, cracks will form. More surface cracks will result to larger spalling and eventually lead to deterioration.

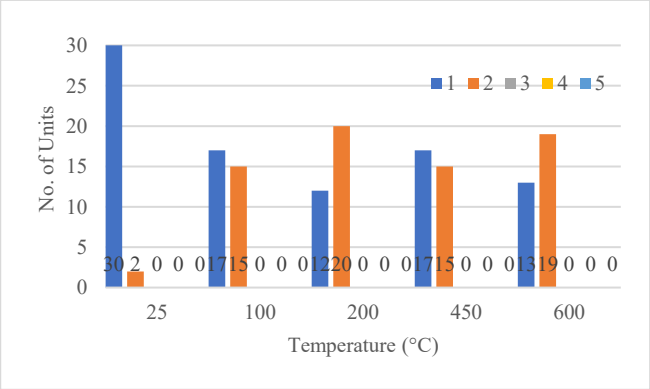
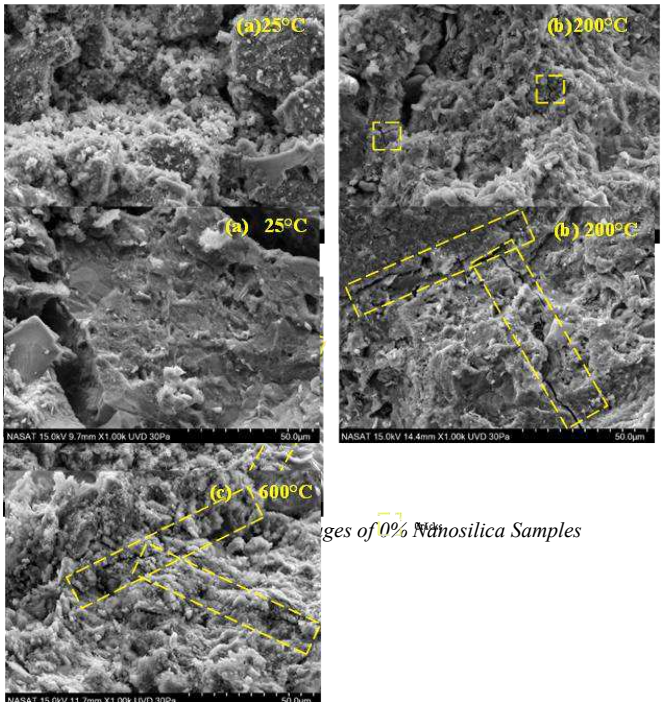


Figure 6. Spalling Severity vs Temperature of varying Nanosilica

Spalling is not a good indicator or tool in assessing the damages in the concrete since most of the samples showed levels 1 and 2 only despite the strength tests showed reduction of strength down to 23.53% and 43.14% for compression and tension respectively for 3% samples. Spalling does not offer wider range of results to categorize which sample has lower strength with two levels of severity only.

3.5. Scanning Electron Microscope

Macro level, concrete is visible to naked eye as conglomerate of aggregate and cement paste. But at micro level, concrete is an undistinguishable matrix of boundaries and granules.



ges of 0% Nanosilica Samples

Figure 8. SEM Images of 3% Nanosilica Samples

Hydration products. CSH formation is in a reticular pattern of crystals which is highly present in SEM images of 0% samples subjected to 25°C, 200°C and 600°C can be seen below. CSH of nS3-25 compared to nS0-25 are more prominent and strongly crystallized in structure by comparing both figures.

Nanosilica creates more surface area to react with free water occupying voids and creating a denser matrix, thus creating a more 'structured' concrete. Calcium hydroxide is distinguishable in both sets of figures as prominent scale-like structure but the sulfates (primarily ettringites) are small to very few as observed in both nanosilica content.

Cracks. For temperature above 200°C, prominent cracks start to inhibit within the matrix until the temperature reaches 600°C compared to the 25°C as seen on and. Samples with 0% nanosilica, are observed to have small discontinuous cracks especially at the 200°C sample. Samples with 0% nanosilica under 600°C has the same discontinuous cracks but are longer in dimension. But samples with 3% nanosilica under 200°C and 600°C both have long continuous cracks. The products of hydrated nanosilica provided the fillers

Table 7. Distribution of Calcium, Silicon, and Magnesium in SEM Samples

			Calcium		Silicon		Magnesium		Total	
Sample No.	Nanosilica	Temp (°C)	Mass Norm. (%)	Atom (%)	Mass Norm. (%)	Atom (%)	Mass Norm. (%)	Atom (%)	Mass Norm. (%)	Atom (%)
1	0%	25	79.46	72.62	17.59	22.93	2.95	4.45	100.00	100.00
2	0%	200	75.33	67.68	21.20	27.18	3.47	5.13	100.00	99.99
3	0%	600	79.51	72.64	17.26	22.50	3.23	4.86	100.00	100.00
4	3%	25	72.55	64.66	25.30	32.17	2.16	3.17	100.01	100.00
5	3%	200	77.91	70.80	19.33	25.07	2.75	4.13	99.99	100.00
6	3%	600	75.12	67.62	22.75	29.26	2.10	3.12	99.97	100.00

between gaps and voids but are weak enough that temperatures above 200°C splits the fillers or medium apart.

4. Conclusion

When high strength concrete is subjected to elevated temperature at 100°C free and adsorbed water evaporates then at 200°C, capillary water evaporates resulting to more adsorbed water loss. Furthermore, at 450°C, aggregates will expand but cement paste will shrink due to loss of water in the matrix that will break the bond between cement paste and aggregates then CSH will start to disintegrate at 600 °C. With lower permeability due to secondary formation of CSH and relatively smaller particle size of nanosilica, concrete will exhibit higher water vapor pressure inside. If the vapor pressure exceeded the net tensile strength of the concrete, cracks will start to form and eventually lead to spalling of concrete layers. Thus, exposing steel or tendons embedded to the matrix increasing risk in further damage and collapse.

High strength concrete has shown stability in compression and tensile strength tests when subjected to temperatures less than 200°C, retaining the lowest residual compressive and tensile strength of 94.31% and 72.22% (both 4% nanosilica), respectively. If subjected to 450°C to 600°C residual compressive and tensile strength retained lowest at 23.53% and 43.14% (both 3% nanosilica), respectively. The addition of nanosilica produced more CSH gels, filled voids and interconnected components within the matrix. But when CSH is exposed to 450°C, more CSH portions break down and disintegrate. Components that are recently in contact won't be able transfer or connect the stress to its neighboring components creating more stress thus lowering strength.

Degradation in compressive strength reached up to 117.4 kPa per °C for 3% samples while control samples only have 11.0 kPa per °C. Moreover, tensile strength reduction reached up to 4.3 kPa per °C for 3% sample compared to 3.0 kPa per °C of control samples. The increase in strength of nanosilica samples ($T \leq 200°C$) is attributed to the stability of CSH due to its development in volume. But a gradual decrease in strength ($T \geq 200°C$) is observed in all samples with nanosilica. Addition of nanosilica imposed higher rate of degradation (both in compression and tension) compared to control. Numerous researches have demonstrated the capability of nanosilica in improving the products of hydration and its effects to concrete but this research has observed that elevated temperature will impose undesirable effects to high strength concrete with nanosilica.

Moisture loss increased up to 0.0161% per °C for 3% compared to 0.0146% per °C of 0% samples. More water is retained inside the concrete due to denser matrix created by addition of nanosilica. With more water-rich interlayers trapped inside, more water will evaporate. Total of 89 samples with no to little spalling (level 1) and small chips (level 2) were found on 71 samples. No samples spalled beyond level 3. Spalling does not offer wider range of results to classify which sample has lower strength with two levels of severity only. Thus, moisture loss is a preferred tool for indicating loss of strength than spalling since concrete is dependent on water for its strength and if water is loss after hydration, it means that concrete has been disturbed.

Under SEM, morphology of nanosilica samples developed block-like medium with long continuous cracks while control samples are craterous in nature with short discontinuous cracks. Microcracks are apparent beyond 200°C for both nanosilica and control samples. The products of hydrated nanosilica provided the fillers between gaps and voids but are weak enough that temperatures above 200°C splits the fillers or medium apart.

These results will be useful in evaluation of post-heated concrete structures such as concrete floors and pads of for boilers, reactors and heat exchangers, including the development of prestressed concrete pressure vessels of nuclear power plants, concrete lining for oil wells and industrial concrete walls. Likewise, these results will help in developing heat suppression additives to concrete to prevent further damages from heat exposure. With these ideas, the paper will help develop further studies of HSC under fire.

5. Recommendations

Compressive and tensile behaviors are still unknown for temperatures beyond 600°C. It is highly recommended to continue the study for temperatures up to 1250°C as fire models of Eurocode 2 and ASTM E119 suggest. To fully understand the behavior of high strength concrete, it is also recommended to study the effects of elevated temperatures to flexural strength of the samples. Similarly, simultaneous heating and load testing will be a great area to ponder to. Correspondingly, to further emphasize the effects of elevated temperature to concrete's microstructure, mercury intrusion porosimetry (MIP) is recommended to be conducted. MIP will be a great step to understand concrete's morphology especially its pore sizes, distribution and volume. Porosity will be a better tool in justifying strength loss or gain of concrete.

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