

## Spalling behaviour of nano SiO<sub>2</sub> high strength concrete at elevated temperatures

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**Abstract.** The behaviour of high strength concrete exposed to high temperature and incorporating Nano SiO<sub>2</sub> and Micro SiO<sub>2</sub> is presented. An experimental programme was developed and carried out which involved testing of Nano SiO<sub>2</sub> and Micro SiO<sub>2</sub> incorporated high strength concrete specimens exposed to temperatures ranging from room temperature to 800 °C. Maximum spalling was noted in Micro + Nano silica specimens. However, the maximum strength loss and temperature induced cracking was noticed in Nano silica specimens.

### 1. INTRODUCTION

Nowadays sustainability development strategies are gaining importance and special attention is being given in applying the sustainable methodologies in designing and production of various products. On the same lines concrete production process is being modified. Concrete, now days can be regarded as the one of the few materials famous for its ubiquitous influence. Concrete is the most commonly used material for construction and its design consumes almost the total cement production in the world. The gradual development of concrete technology has promoted the use of high strength concrete (HSC). Concrete strengths much higher than 60 MPa are gaining acceptance in the construction industry. A race for making High Strength Concrete has evolved throughout the construction arena and different materials are being incorporated in conventional concrete to make the process of developing HSC easier and flawless.

From the early studies on High strength concrete it has been experienced that HSC provides a high level of structural performance, especially in strength and durability, compared to traditional, normal-strength concrete (NSC). The HSC was earlier used in special structures but as of now HSC has found its way in many projects. Although one of the basic materials for producing this type of concrete is silica fume because it enhances the concrete's mechanical properties, more recently researchers have reported that nanosilica has superior performance to silica fume in enhancing the compressive strength of concrete [1–3]. Addition of 10% nano-SiO<sub>2</sub> with dispersing agents was observed to increase the compressive strength of cement mortars at 28 days by as much as 26%, compared to only a 10% increase with the addition of 15% silica fume [4]. Nano-SiO<sub>2</sub>, additionally, was shown to accelerate the hydration reactions of both C3S and an ash–cement mortar as a result of the large and highly reactive surface of the nanoparticles [5]. It has been found that nanosilica particles not only increase the material's hydration but it also actively raises the pozzolanic activity rather than behaving exclusively as a filler for the calcium silicate hydrate [3, 6]. In this process it makes the concrete structure more compact and dense. The permeability of the nanosilica concrete is highly reduced and the concrete formed is more durable.

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Another important issue regarding the production of HSC is that the use of large quantities of cement produces increasing CO<sub>2</sub> emissions, and as a consequence the green house effect. A method to reduce the cement content in concrete mixes is the use of silica fines. One of the silica fines with high potential as cement replacement and as concrete additive is nano-silica (nS). Nano silica is being used as a replacement of cement in production of high strength concrete (HSC). Nano silica reacts with calcium hydroxide (CH) to develop more of the strength carrying structure of cement: calcium silica hydrate (CSH). The concrete incorporating nano silica is denser than ordinary concrete or micro silica concrete; helps achieve greater strengths and increases impermeability. Studies conducted have shown that the 28 day compressive strength of Nano SiO<sub>2</sub> concrete is significantly higher than the ordinary concrete [6].

In the concrete structures exposed to high temperatures, concrete spalling may occur and can destroy the part of the structure and/or reduce the load carrying capacity of the structure substantially. The effect of high temperature on the concrete structures has been well studied by the researchers. Many studies on the effect of elevated temperatures upto 1100 °C on the concrete have been documented. Spalling is regarded as one of the complex and poorly understood phenomenon in the reaction of concrete to elevated temperatures. It therefore needs a due consideration when designing a concrete structure for fire. However not much literature is available regarding the behaviour of Nanosilica High strength concrete exposed to elevated temperatures. In this paper the influence of high temperature on Nano SiO<sub>2</sub> based HSC was studied. The main objectives of the study were: (i) to develop a HSC with Nano SiO<sub>2</sub>, (ii) to find out the effect of higher temperatures on the relative strength loss, mass loss on the Nano SiO<sub>2</sub> based HSC, (iii) and ultimately to study its spalling behaviour.

## 2. RESEARCH SIGNIFICANCE

Nano SiO<sub>2</sub> based HSC is a new entry in the world of concrete technology and has potential to replace the conventional HSC. A better understanding of its behaviour under different conditions will help build confidence in its applicability. This study provides the initial findings on the behaviour of Nano SiO<sub>2</sub> HSC at elevated temperatures.

## 3. EXPERIMENTAL PROGRAMME

### 3.1 Materials

Ordinary Portland cement from a single lot was used in the experimental investigation. The cement conformed to Indian standard IS: 8112-1989 [7]. The micro silica having a silica content of more than 92 percent conforming to IS: 15388-2003 [8] was used in the investigation. Amorphous nanosilica powder was procured from Singhal Exports. The properties of the Micro and Nano silica are tabulated in Table 1. The fine aggregate used in this study was locally available river sand with a fineness modulus of 2.31 and was passed through a sieve of 4.75 mm. The coarse aggregate was crushed stone aggregate of maximum nominal size of 10 mm. The superplasticizer used was modified polycarboxylic ether type commercially called Glenium ACE 30.

### 3.2 Methods

#### 3.2.1 Mixture Proportions

Three different mixes with 5% Nano SiO<sub>2</sub>, 10% Micro SiO<sub>2</sub>, and with 5% each of both Nano as well as Micro SiO<sub>2</sub> were designed in the study. The mixes were designed for a target strength of 80 MPa. While designing the mixes it was noted that the concrete made with Nano SiO<sub>2</sub> demonstrated very low consistency and higher water was required for the target slump. Consequently both the water content as

**Table 1.** Physical and chemical Characteristics of Nano SiO<sub>2</sub> and Micro SiO<sub>2</sub>.

Property	NanoSiO <sub>2</sub>	Property	MicroSiO <sub>2</sub>
Type	1.2-1.6, amorphous, non porous	Blaines's fineness	22000 cm <sup>2</sup> /g
Purity	99.5%	Specific gravity	2.20
APS*	16–20 nm	SiO <sub>2</sub>	95.1%
SSA*	170–200 m <sup>2</sup> /g	Loss on ignition,	2.79
Colour	white	percent by mass	
Bulk Density	< 0.10 g/m <sup>3</sup>		
True Density	2.4 g/m <sup>3</sup>		

\* SSA: specific surface area; APS: Average particle size.

**Table 2.** Mix proportions and compressive strengths of the test concrete mixes.

	NanoSiO <sub>2</sub> HSC (NHSC)	Micro SiO <sub>2</sub> HSC (MHSC)	Nano-Micro SiO <sub>2</sub> HSC (MNHSC)
Cement, Kg/m <sup>3</sup>	600	600	600
Micro SiO <sub>2</sub> , Kg/m <sup>3</sup>	—	60 (10%)	30 (5%)
Nano SiO <sub>2</sub> , Kg/m <sup>3</sup>	30 (5%)	—	30 (5%)
Coarse Aggregate, Kg/m <sup>3</sup>	1055	1055	1055
Fine Aggregate, Kg/m <sup>3</sup>	625	625	625
Water, Kg/m <sup>3</sup>	192	168	180
Super Plasticizer, l/m <sup>3</sup>	9	7.5	8
Compressive strength at 28 days, MPa	83	85	89
Slump, mm	93	97	95

well as the superplasticizer dosage was higher for the Nano SiO<sub>2</sub> HSC. The mix proportions of all the three types of mixes are given in Table 2.

### 3.2.2 Casting and curing

A total of 36 cube specimens were cast and tested under this investigation. They included 12 numbers of Nano silica concrete specimens, 12 numbers of micro silica concrete specimens and 12 specimens were cast with both Nano as well as Micro SiO<sub>2</sub>. The mixing of the ingredients was done in a tilting mixer and care was taken in handling the NanoSiO<sub>2</sub> powder as it was observed that most of the powder dusts up as soon as the mixing of dry mix is started. The problem was solved by making the mix little wet before the Nano SiO<sub>2</sub> was being added. All the specimens were cubes with a size of 100 mm × 100 mm × 100 mm. The specimens were removed from the moulds after 24h and were cured for 14 days in curing tanks. They were taken out from the curing tank after 14 days and were kept inside the laboratory for another 14 days. This was done to simulate the field conditions and also to ensure that the moisture content remains as low as possible so that the spalling due to high moisture content is avoided. The specimens were cast and tested in triplicate in order to get the average of three results thus making a set of 4 nano silica, 4 micro silica and 4 Nano-Micro silica concrete samples.

Before exposing the specimens to different elevated temperatures the mass and ultrasonic pulse velocities for all the specimens was measured.

**Table 3.** Specimen nomenclature and exposure scheme.

Specimen	Temperature	Micro/Nano Silica	Specimen	Temperature	Micro/Nano Silica
MS1, MS2, MS3	200 °C	Micro Silica	NS7, NS8, NS9	800 °C	Nano Silica
MS4, MS5, MS6	500 °C	Micro Silica	NS10, NS11, NS12	Ambient	Nano Silica
MS7, MS8, MS9	800 °C	MicroSilica	MN1, MN2, MN3	200 °C	Micro + Nano Silica
MS10, MS11, MS12	Ambient	Microsilica	MN4, MN5, MN6	500 °C	Micro + Nano Silica
NS1, NS2, NS3	200 °C	Nano Silica	MN7, MN8, MN9	800 °C	Micro + Nano Silica
NS4, NS5, NS6	500 °C	Nano Silica	MN10, MN11, MN12	Ambient	Micro + Nano silica



(a)



(b)

**Figure 1.** (a) Programmable muffle furnace. (b) Specimens after exposure.

### 3.2.3 Fire resistance

The specimens were exposed to 4 different temperature regimes viz: room temperature (27 °C), 200 °C, 500 °C, and 800 °C in a programmable muffle furnace (Fig. 1). The exposure scheme of the specimens is shown in the Table 3. The average moisture content (% by mass) at the time of exposure was 2.4% in MS specimens, 2.7% in NS specimens and 3.6% in MN samples. The temperature was increased at a rate of 5 °C/min and the target temperature was maintained for 2 hours after which the specimens were allowed to cool to room temperature.

### 3.2.4 Mass and ultra sonic pulse velocity measurements

Ultra sonic pulse velocity measurements were used to evaluate the induced damage in concrete. The tests are widely recognized as standard tests for condition assessment of concrete structures. In the present study each specimen was weighed and the ultrasonic pulse velocity was measured for each specimen before exposing them to elevated temperatures and also after exposure.

### 3.2.5 Compressive strengths

The specimens which survived the spalling were tested for compressive strength in a 200T compression testing machine and the results compared with the specimens at room temperature.

#### 4. RESULTS AND DISCUSSION

The damage to the concrete after exposure to elevated temperatures was assessed on the basis of visual inspection. It usually includes visual observations of color change, cracking and the spalling of the concrete surface [9]. Figures 2(a–f) illustrate the appearance of concrete specimens after exposure to varied temperature regimes. There was no visible effect on the surface of any of the specimens, which were exposed to a temperature of 200 °C. However the specimens which were exposed to a temperature of 500 °C showed varying amounts of cracking and spalling. The Micro SiO<sub>2</sub> specimens showed some fine surface cracks, while the Nano SiO<sub>2</sub> specimens showed significant cracks. In MN specimens the cracking was quite evident and one of the specimens even spalled. The MS specimens which were exposed to 800 °C had cracks on the surface and the concrete had started to deteriorate. On the contrary in NS specimens the cracking was very extensive at 800 °C temperature and the cracks went deep into the cross section of the specimens [Fig 2(b)]. The concrete was observed to be loosely bound and the concrete chips would come out on just minor disturbance with hands. In case of MN samples the spalling took place in two of the specimens at 800 °C temperature, but the cracking was not too extensive. The results were in contradiction with what we had anticipated. It was thought that the NS samples would spall more but in the present study the MN samples spalled more. The reason could be extra dense micro structure due to the combined effect of Micro and Nano SiO<sub>2</sub> in MN samples. The particles of Nano SiO<sub>2</sub> would have filled the voids which remain after the Micro SiO<sub>2</sub> particles react with the calcium hydroxide and thus rendering the microstructure of MN specimens dense. Also the MN specimens showed greater water retention properties and this led to build up of higher pore pressure and ultimately more chances of spalling. Nevertheless the NS samples cracked extensively indicating the poor thermal behaviour of NS HSC.

The effect of elevated temperature on the weight loss of different specimens is shown in the Figure 3. In each concrete mix the losses increased with increase in temperature. These losses were about 3.17%, 7.53% and 10.4% respectively at 200 °C, 500 °C and 800 °C in NS samples. In case of MS specimens the mass loss was 2.47%, 5.47% and 14.28% respectively and in case of MN samples the loss in mass was reported as 2.88%, 10.79% and 19.45% respectively at 200 °C, 500 °C and 800 °C. The mass loss in case of MN samples was highest at all the temperatures. The mass loss was due to spalling in MN samples due to higher initial moisture content.

Figure 4 shows the variation in loss of compressive strength of the specimens at different elevated temperatures. After exposure at 200 °C, all the specimens of different mixes showed an increase in the compressive strength, the highest (10.44%) being in NS specimens. The increase of strength in MN and MS specimens was almost same. It is to be noted that exposing the specimens to a temperature to a tune of 200 °C can increase the hydration process by producing more high-density calcium silicate hydrate and by increasing the reactivity of nanosilica, which enhances the strength [10]. Nanosilica converts more of the calcium hydroxide to calcium silicate hydrate and this process gets accelerated for the concrete exposed upto such temperature. However on further increasing the exposure temperature the specimens showed loss in compressive strength which increased on increasing the temperature to 800 °C. A loss of 79.12% was found in NS specimens, 48.63% in MS and 71.9% in MN specimens. Such behaviour can be attributed to excessive build up of vapour pressure which led to extensive cracking in NS samples. One of the peculiar behaviour of NS specimens was that during compression testing the cubes crushed into burnt powdery substance which showed very less cohesion and more like burnt clay.

The loss in UPV as shown in Figure 5 also depicts the degradation of concrete as the exposure temperature was increased. The UPV increased upto 200 °C thereafter showing a decreasing trend with increase in temperature. Again the degradation was more in NS specimens. A loss of about 82% in UPV was measured in concrete with Nano SiO<sub>2</sub> while the MS specimens showed a loss of only 60% in UPV. In case of MN specimens the UPV loss was found to be 74.72%. Such behaviour may be again attributed to extensive cracking in NS specimens due to build up of high vapour pressure.



(a) MN specimens after 800 °C



(b) NS specimens after 800 °C



(b) MS specimens after 800 °C



(d) MS specimens after 500 °C



(e) MN Specimens after 500 °C



(f) NS specimens after 500 °C

**Figure 2.** Specimens after exposure to different temperatures.

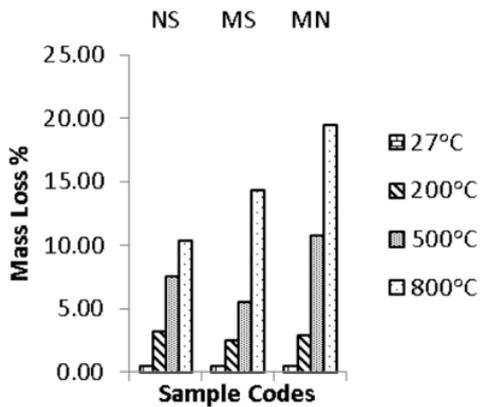


Figure 3. Mass loss at different temperatures.

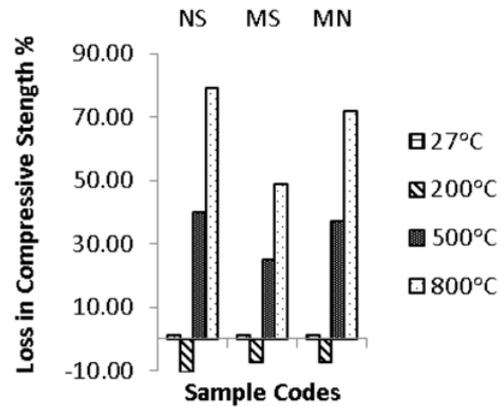


Figure 4. Variation in compressive strength loss.

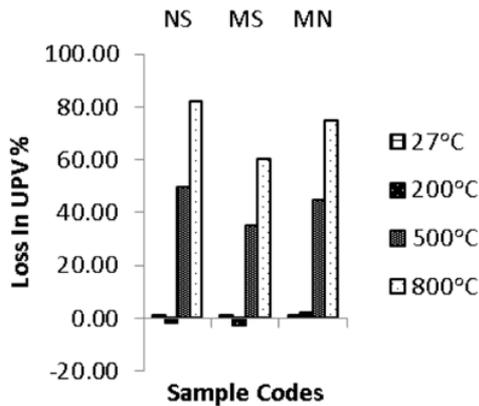


Figure 5. Loss in UPV of different specimens.

## 5. CONCLUSIONS

Experimental study was conducted on High strength concrete, incorporating Nano SiO<sub>2</sub>, Micro SiO<sub>2</sub> and Nano + Micro SiO<sub>2</sub>, subjected to elevated temperatures. Following conclusions may be drawn from the study:

1. Micro+Nano silica specimens retain more moisture than Nanosilica and Microsilica specimens.
2. Maximum spalling was noted in Micro+Nano silica specimens and very less spalling in was observed in Nanosilica and Microsilica specimens.
3. The maximum thermal damage and temperature induced cracking was noticed in Nano silica specimens. Although the Micro + Nanosilica specimens showed spalling but very less thermal damage and cracking was observed in these specimens.
4. The maximum compressive strength loss was found in Nanosilica specimens. The Nano SiO<sub>2</sub> is more vulnerable to high temperature and damage and loss in strength, mass and quality was found to be more extensive in this type of concrete.
5. It was noted that the workability of Nano SiO<sub>2</sub> concrete is highly reduced and the Nano SiO<sub>2</sub> mixes have more water demand. The percentage of Nano SiO<sub>2</sub> used was 5% and was very high which increased the water demand and also most of the nanosilica may not have reacted with the

hydration products which further needs to be investigated. It is also worth a mention that the Nano SiO<sub>2</sub> was in amorphous powdery state which makes it very difficult to mix properly.

6. Extensive testing is underway and further inputs on the results will be displayed in the workshop.

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