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Mechanical properties and microstructure of high strength concrete containing polypropylene fibres exposed to temperatures up to 200 °C

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Abstract

High strength concrete has been used in situations where it may be exposed to elevated temperatures. Numerous authors have shown the significant contribution of polypropylene fibre to the spalling resistance of high strength concrete. This investigation develops some important data on the mechanical properties and microstructure of high strength concrete incorporating polypropylene fibre exposed to elevated temperature up to 200 °C. When polypropylene fibre high strength concrete is heated up to 170 °C, fibres readily melt and volatilise, creating additional porosity and small channels in the concrete. DSC and TG analysis showed the temperature ranges of the decomposition reactions in the high strength concrete. SEM analysis showed supplementary pores and small channels created in the concrete due to fibre melting. Mechanical tests showed small changes in compressive strength, modulus of elasticity and splitting tensile strength that could be due to polypropylene fibre melting.

Keywords: Thermal treatment; Hydration products; SEM; Compressive strength; Mechanical properties; High performance concrete

1. Introduction

High strength concrete offers various benefits derived from its higher strength and stiffness, and for the last few years, the use of high strength concrete has become increasingly popular. A greater understanding of its behaviour under different conditions will improve confidence in its use. As the use of high strength concrete becomes common, the risk of exposing it to elevated temperatures also increases. In order to predict the response of structures employing high strength concrete during and after exposure to elevated temperatures, it is essential for the microstructural properties of high strength concrete subjected to elevated temperatures to be clearly understood.

For several decades it has been established that the mechanical properties of concrete (normal strength concrete) are modified with high temperature exposure [1–3]. Results of many recent high temperature exposure tests have shown that there are significant differences between the performance of high strength concrete at elevated temperature compared with normal strength concrete [4–8]. A comprehensive compilation of experimental results on the mechanical properties of concrete when exposed to rapid heating was presented by Phan and Carino [9]. The amount of test data available on high strength concrete exposed to high temperature points out the number of variables (concrete strength, concrete age, concrete density, concrete water content, aggregate type, addition of silica fume and/or fibre, test conditions, specimen size, heating rate, etc.).

Published data indicate that silica fume concrete, when exposed to temperatures up to 300 °C, develops more spalling than normal concrete [4,10–12]. Thermal stresses and pore pressure in heated concrete members were studied in order to understand concrete spalling [13–16]. Unfortunate combinations of low permeability, low porosity, low thermal transmission and high moisture content were supposed to lead to increased tendency to spalling. To prevent such problems, different ways in which the high

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temperature resistance of high strength concrete can be improved were investigated. The manner in which different fibres affect fire properties of mortar has been investigated by Sarvaranta et al. [17] and Sarvaranta and Mikkola [18]. The results showed that fibre type affects both heat and mass transfer, as well as the extent of spalling of the mortar at elevated temperatures. Lie and Kodur [19] studied thermal and mechanical properties of steel fibre reinforced concrete at elevated temperatures. They showed that the effect of steel fibres on the mechanical properties is greater than that on the thermal properties of the tested concretes. They concluded that concrete heat resistance could be performed by incorporating steel fibres. This is contrary to Hertz [11] whose results showed that the presence of steel fibres in concrete does not reduce the risk of spalling.

In the last 7 years, due to higher pore pressure in high strength concrete compared with normal strength concrete, one idea was to integrate artificial pores or channels into the high strength concrete matrix, in which the developing water vapour pressure can be relieved to a level similar to normal concrete with sufficient capillary pores. The use of LMPF (low melting point fibre) in high strength concrete began to be investigated. Some authors carried out and reported on comprehensive investigations on the effects of elevated temperatures on their mechanical and microstructural properties. Diederichs et al. [20] and Nishida et al. [21] carried out experiments on polypropylene high strength concrete at elevated temperatures. The likelihood of spalling due to thermal exposure was reduced for the high strength fibre concretes when compared to the reference high strength concrete. They showed that deleterious spalling can be greatly reduced by adding to the concrete small quantities (on the order of 0.1% by volume) of fibres made from a low melting-point polymer. These results generally agree with those obtained by Hoff [22] and Bilodeau et al. [23] on fire resistance of high strength concretes incorporating synthetic fibres for offshore concrete platforms. Breitenbücker reported that high strength concrete incorporating polypropylene fibres was applied the first time in Frankfurt in 1995 [24]. The 115 m high “Japan centre” was built using high strength concrete (HSC 105) with high fire-resistance for several structural members. The polypropylene fibres dosage was 2 kg/m^3. The results of the French National Project BHP2000 [25] indicated that the incorporation of polypropylene fibres in high performance concrete had a significant effect on its hydraulic behaviour at high temperature. By adding polypropylene fibres to concrete the water vapour pressure at high temperature was significantly decreased. The optimal dosage of fibres was found to be close to 1.5 kg/m^3.

A mathematical and computational model to simulate the two-dimensional thermal response of high strength concrete columns subjected to high temperature was presented by Ahmed and Hurst [15]. Results from parametric studies emphasised the importance of performing thermophysical material property tests under high temperature exposure conditions similar to those at which full-scale specimens are to be tested. Some experimental results were included for comparison with model predictions.

Many research studies have been also examined the hygrothermal consequences following a loss of coolant accident (LOCA) on a nuclear containment vessel. The accident conditions consist of a rise from ambient to a maximum temperature of 160 °C and a pressure of 650 kPa. This rise is followed by a dwell and a cooling that lasts several days [26]. Kuznetsov and Rudzinskii [27] studied high temperature heat and mass transfer in a concrete layer used for biological protection of nuclear reactors at critical heat loads. Kontani and Shah [28] published details on the pore pressure and temperature distribution in concrete at a sustained high temperature (171 °C) following a loss of coolant accident. It is admitted that, in the case of an accident, the temperature inside the concrete containment vessel may increase but may not exceed 180 °C. Compared with fire standard curve the concrete heating rate is very low.

The main objective of this investigation was to study the effect of elevated temperature on properties of two concretes intended for nuclear applications: one high strength concrete incorporating polypropylene fibres and one high strength concrete without fibres. The applied heating curve was not the standard fire curve but a heating–cooling cycle close to RILEM recommendations [29]. The study adds important data to existing information on the behaviour of high strength fibre concrete under elevated temperatures.

2. Test program

The test specimens were subjected to 200 °C, and the behaviour compared to that observed at 20 °C. During the heating period moisture in the test specimens was allowed to escape freely. The tests were carried out on 160 × 320 mm and 110 × 220 mm concrete cylinders. Two mixtures containing 20 mm maximum size aggregate were tested. Normal Portland cement, French CPA CEM I 52.5, was used. A sulfonated naphthalene formaldehyde condensate type superplasticizer was used. The silica fume was not used as a replacement, but as an addition, to the cement. The

Table 1
Mixture proportions of both concretes

Materials	B1 control high strength concrete	B3 high strength concrete with polypropylene fibre
Gravel 12/25	765	739
Gravel 5/12	298	288
Sand 0/5	734	709
Cement CPA CEM I 52.5	375	408
Silica fume	38	41
Water	141	136
Superplasticizer	11.3	10.9
Fibres		1.8
w/cm	0.34	0.30

Table 2
Density of the both tested concretes

	B1 concrete	B3 concrete
Density prior to heating at 200 °C	2.36	2.33
Density after heating at 200 °C	2.23	2.20

polypropylene fibres were 13 mm long, and the fibre dosage was fixed to 1.8 kg/m^3. Table 1 gives the mixture proportions of both concretes.

The aggregates, cement and silica fume were first mixed dry (fibres were added in B3 concrete) for 2 or 3 min, then water mixed with superplasticizer was added to the mixture. Mixing continued for an additional 3 min. All cylinders were cast in two layers in cardboard moulds and were compacted by using a vibrating table. The specimens were capped with plastic sheet. Plastic sheet in the interior part of the cardboard mould and plastic caps sealed the specimens to ensure mass curing. The specimens were then transferred to the moist-curing room until required for testing. Prior to and after exposure of the specimens to elevated temperatures the densities were determined.

The heating equipment was an electrically heated kiln. The specimens were positioned in the kiln in a fashion which minimised variation of temperature between specimens. Temperatures at the centre and at the surface of the specimens were monitored by type K thermocouples connected to a data acquisition unit.

Cylinders from each mixture were placed in the kiln and the kiln was heated to the desired temperature of 200 °C at a rate of 0.5 °C/min. After 3 h at this temperature the kiln was turned off. It was allowed to cool down before the specimens were removed to prevent thermal shock to the specimens. The rate of cooling was not controlled. The tests to determine compressive strength were made according to NF P 18-406 French specifications [30]. The data were obtained for an age of 62 days up to 92 days. At least three specimens were tested for each variable. The microstructure of both concretes was analysed with the help of DSC, TG and SEM.

3. Results and discussion

3.1. Density

The initial density of B3 concrete (polypropylene high strength concrete) was less than that of B1 concrete (high strength concrete without fibre). Density decrease of B3 concrete was similar to that of B1 concrete (Table 2). The weight change of concrete was mainly due to the dehydration of cement paste. The weight of the melted fibres was negligible. During heating the temperature values indicated that heat transfer through B3 concrete was lower than that through B1 concrete used as reference.

3.2. Mechanical properties

3.2.1. Initial compressive strength and modulus of elasticity

In order to asses the effect of elevated temperatures on concrete mixes under investigation, measurements of properties of test specimens were made shortly before and after heating, when specimens were cooled down to room temperature. The initial strength of heat-test specimens was determined on companion reference specimens among which a set of cylinders for each type of concrete subjected to heat exposure. Reference test specimens were crushed at the beginning of the heating tests. The results of these measurements are shown in Fig. 1 as B1-20 °C and B3-20 °C. The modulus of elasticity of the B1 concrete was close to that of B3 concrete although the compressive strengths were different. As can be seen the increase of the cement content had less effect on the modulus of elasticity than on the compressive strength.

3.2.2. Residual compressive strength and modulus of elasticity

The changes in mechanical properties in the series of concrete made with the same cement, silica fume and sand but different cement contents were also studied after exposure to 200 °C. The behaviour of the tested specimens

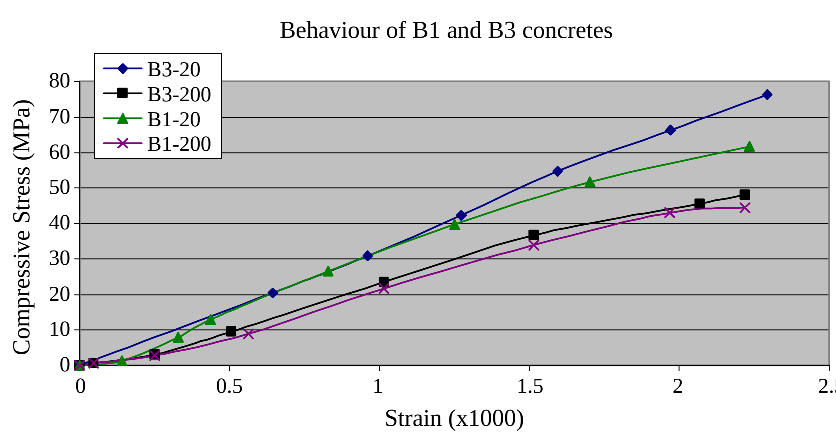

Fig. 1. Stress–strain relationships of B1 and B3 concretes at room temperature (20 °C) and after exposure at 200 °C and cooled down.

Table 3
Compressive strength and modulus of elasticity of the tested concretes

	B1	B3
Initial compressive strength (MPa)	61 (100%)	76 (100%)
Compressive strength (MPa) (200 °C)	44 (72%)	48 (63%)
Initial modulus of elasticity (GPa)	28 (100%)	33 (100%)
Modulus of elasticity (GPa) (200 °C)	20 (71%)	22 (67%)

is presented in Fig. 1. The results of modulus of elasticity and compressive strength are summarised in Table 3.

After initial heating up to 200 °C, both the compressive strength and modulus of elasticity were reduced (to 28–37% of the non-heated strength and 29–33% of the non-heated modulus of elasticity). The results indicated that in the temperature range tested the mechanical properties of the B3 concrete decreased more than that of the B1 concrete.

3.2.3. Initial and residual splitting tensile strength

The specimens which were used for splitting tests were 110 × 220 mm cylinders. The results are shown in Table 4. The heat resistance of the splitting tensile strength appeared to decrease when polypropylene fibres were incorporated into concrete. This is probably due to the additional porosity and small channels created in the mortar by the fibres melting.

3.3. Differential scanning calorimetry

DSC analysis was carried out using the SETARAM Labsys 1200 apparatus. DSC is a measurement method used to determine the heat transformation and the enthalpy change of materials. A sample (cut from mortar paste) is submitted to a control temperature program with constant heating rates or constant temperatures. The measured heat flux to and from the sample indicates the transformation temperature ranges.

When a cementitious material such as concrete is being heated several chemical and physical phenomena occur in the temperature range between 100 and 900 °C. The reactions initiated during the heating of both high strength concretes were studied. The heating rate was 10 °C/min. DSC curves are shown in Fig. 2.

Several physical phenomena occurred in the temperature range between 100 °C and 250 °C: vaporization of water in the cementitious matrix (110 °C), CSH dehydration, fibre shrinkage and melting (170 °C). These results are very similar to those published by Phan and Carino [9]. Two endothermic peaks can be seen in the same temperature regions between 100 and 250 °C. In the DSC curves of both

Table 4
Splitting tensile strength of the tested concretes

	B1	B3
Initial splitting tensile strength (MPa)	3.9 (100%)	4.7 (100%)
Splitting tensile strength (MPa) (200 °C)	2.7 (69%)	2.9 (62%)

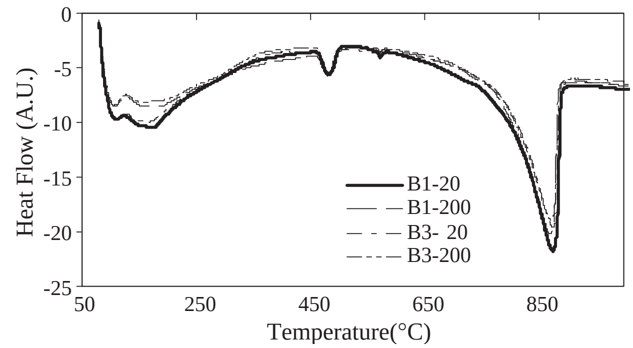

Fig. 2. DSC curves of both high strength concretes.

B1 and B3 concretes the following transformations can be identified:

- water evaporation at 110 °C,
- fibre melting at about 170 °C,
- first stage of CSH dehydration at 170 °C,
- A peak is seen near 480 °C in all the curves. It is probably due to portlandite. Although both B1 and B3 high strength concretes included silica fume that reacts with calcium hydroxide during cement hydration, a non-negligible amount of portlandite is suspected. The observed discrepancy was already noticed in a previous work by Weigler and Fisher [3]. No satisfying explanation has been found.
- Quartz transformation from α rhomboedric shape to β hexagonal shape at 573 °C,
- At about 870 °C a large peak can be seen. It is mainly due to decomposition of calcium carbonate and CSH phases.

One can notice that, after 250 °C, the behaviour of B3 concrete is very close to that of B1 concrete. The effect of fibre is mainly significant in the temperature region from 100 to 250 °C before the entire melting.

3.4. Thermogravimetry

The thermogravimetry analysis was carried out by using the SETARAM Labsys 1200 device. A sensitive balance is used to follow the weight change of the material sample as a function of temperature. The heating rate in air was 10 °C/min. Results are shown in Table 5. Samples were cut from non-heated (20 °C) and heated (200 °C) B1 and B3 concrete specimens.

The initial water content of B1 concrete was close to that of B3 concrete. The weight loss observed in the TG curves occurred in the temperature ranges observed for decomposition reactions seen on DSC curves. As regards weight loss due to heating both B1 and B3 concretes had similar behaviour. It is quite obvious that the C–S–H dehydration between 100 and 450 °C in non-heated concrete was greater than that in heated concrete. The $Ca(OH)_2$ decomposition between 450 and 520 °C seems to

Table 5
Weight losses in reference to the sample initial weight

Reaction	Sample prior test temperature	Weight loss (%)
Water evaporation and C–S–H dehydration, 100–450 °C	B1-20 °C	3.00
	B1-200 °C	1.61
	B3-20 °C	1.99
	B3-200 °C	1.70
$Ca(OH)_2$ decomposition, 450–520 °C	B1-20 °C	0.32
	B1-200 °C	0.34
	B3-20 °C	0.30
	B3-200 °C	0.44
$CaCO_3$ decomposition, 600–900 °C	B1-20 °C	15.53
	B1-200 °C	13.47
	B3-20 °C	12.60
	B3-200 °C	13.64

B1-20 °C means non-heated B1 concrete. B1-200 °C means B1 concrete heated at 200 °C and cooled down prior to TG test.

indicate that both concretes had the same amount of portlandite.

3.5. Scanning electronic microscopy

Non-heated as well as heated specimens were observed by SEM. Fig. 3 shows polypropylene fibres scattered in non-heated high strength concrete. Fig. 4 shows pieces of fibres after melting. At 200 °C polypropylene fibres had lost their solid structure. There was a significant difference between the porosity of B1 and B3 concretes after exposure at 200 °C. When the high strength polypropylene fibre concrete is heated up to 200 °C, fibres readily melt and volatilise, creating additional pores and small channels in the concrete that may act to relieve high internal moisture pressures. SEM analysis showed traces of melted fibres (Fig. 5). Polypropylene fibres decreased in length under heating owing to relaxation. They melted on further heating. Clearly, the use of fibre affects the porosity at high temperature of the high strength concrete. This may decrease the pore pressure inside the high strength concrete. The fibres affect the porosity then the release of moisture from the material.

Fig. 3. Polypropylene fibres scattered in the high strength concrete.

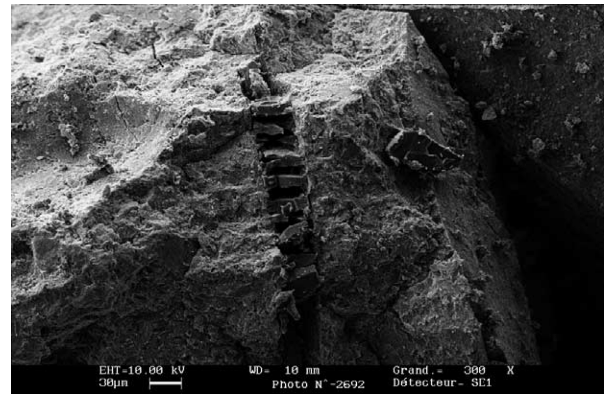

Fig. 4. Pieces of fibres after melting.

3.6. Discussion

The microstructure controls the water expulsion from the concrete at normal as well as at high temperature. Therefore the pore structure at high temperature may have a considerable influence on the spalling behaviour of the high strength polypropylene fibre concrete.

The melting of polypropylene fibres may be beneficial to the behaviour of fibre high strength concrete under thermal exposure. In case of intense high temperature exposure, not all water is expelled fast enough from the high strength concrete. This will result in vaporisation at higher temperatures and the creation of high pressures inside the paste [10–12]. The additional porosity and small channels created by the melting of polypropylene fibre may lower internal vapour pressures in the concrete, and reduce the likelihood of spalling. The microstructural behaviour may of course be affected by dimensions and amount of fibre.

4. Conclusion

This investigation was carried out to develop data on the effect of elevated temperature up to 200 °C on properties of two concretes intended for nuclear applications. A high

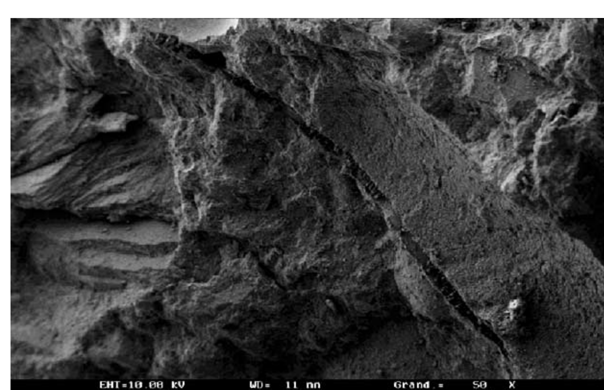

Fig. 5. Traces of melted fibres in high strength concrete.

strength concrete incorporating polypropylene fibres and a high strength concrete without fibres were investigated. Mechanical properties of concrete were studied at room temperature and after exposure at 200 °C. The addition of polypropylene fibres (1.8 kg/m^3) may lead to small changes in residual compressive strength, modulus of elasticity and splitting tensile strength due to fibres melting during heating. The heat resistance of the mechanical properties appeared to decrease when polypropylene fibres were incorporated into concrete.

The microstructure of the both tested concretes was examined with the help of TG, DSC and SEM. Thermogravimetry and differential scanning calorimetry analysis showed little difference between the two tested concretes. The temperature ranges of the decomposition reactions were very definitely similar. Scanning electron microscopy gave clear indications of the fibre melting and supplementary porosity creation. There was a significant difference between the porosity of polypropylene fibres high strength concrete and the reference high strength concrete after exposure at 200 °C. This may result in lower vapour pressure in the polypropylene fibres high strength concrete in the early stage of heat exposure. It means lower risk of concrete spalling in case of accident.

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