Development of ultra-high performance concrete with high fire resistance

Xiangwei Liang [a],*, Chengqing Wu [a], Yu Su [a], Zhu Chen [b], Zhongxian Li [b]

[a] *Centre for Built Infrastructure Research, School of Civil and Environmental Engineering, University of Technology Sydney, NSW 2007, Australia*

[b] *Tianjin Key Laboratory of Civil Structure Protection and Reinforcement, Tianjin Chengjian University, Tianjin 300384, China*

Abstract

Fire or high temperature is a big challenge to ultra-high performance concrete (UHPC). Strength loss of UHPCs can reach up to 80% after exposure to 800 °C. In this study, a total of six UHPC mixtures were designed and tested after subjected to elevated temperatures up to 1000 °C. The effects of aggregate type, fibre type and heating rate were investigated. Residual compressive strengths and stress-strain relationships were studied. Besides, attention was paid to explosive spalling since UHPCs are usually of compact structure and thus more vulnerable to explosive spalling than other concretes. Scanning electron microscope (SEM) analysis was conducted to help understand the mechanism of variation of internal structure under different temperatures. It was found the mixture containing steel slag and hybrid fibre had excellent fire resistance. After being subjected to 1000 °C, this mixture retained a residual compressive strength of 112.8 MPa or a relative value of 69%.

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* Corresponding author.
  E-mail addresses: xiangwei.liang@student.uts.edu.au (X. Liang), chengqing.wu@uts.edu.au (C. Wu).

1. Introduction

Due to rampant terrorism, governments and researchers are paying growing attention to protective structures against blast and fire. UHPC is considered a desirable material for this kind of structures. The Portland Cement Association (PCA) defines UHPC as "a high-strength, ductile material formulated by combining portland cement, silica fume, quartz flour, fine silica sand, high-range water reducer, water, and steel or organic fibers. The material provides compressive strengths up to 29,000 pounds per square inch (psi) and flexural strengths up to 7,000 psi". By contrast, normal strength concrete (NSC) and high performance concrete (HPC) are much inferior in performance such as strength, toughness, ductility and durability. In the last decades, a lot of effort has been made to improve mechanical properties of UHPC. For example, most recently some nano materials were investigated as a new additive to UHPCs [1, 2]. However, fire or high temperature is still a great threat to all kinds of concretes including UHPCs.

Chan et al. [3] experimentally studied the behaviour of HPC subjected to high temperatures up to 800 °C and they found the compressive strength of HPCs degraded more sharply than NSCs. Similar findings were obtained by [4, 5]. Sobia et al. [6] carried out a comparative study on ultra-high performance fibre-reinforced cementitious composites (UHPFRCC) before and after heat treatment. Their test results revealed the residual compressive strength of UHPFRCC after being exposed to 1,000 °C was around 41% of its room-temperature strength. Much severer deterioration was observed by [4, 7, 8]. Choe et al. [4] suggested the relative residual compressive strength of ultra-high-strength concrete being heated to 1,000 °C was

only 2%. Another threat a fire can pose to UHPCs is explosive spalling and severe explosive spallings were observed by Kahanji et al. [9, 10]. Lin et al. [11] and Akca and Zihnioğlu [12] proposed that increasing pore pressure due to water evaporating and thermal strain were the major reasons for concrete spalling. UHPCs usually suffer more serious spalling than NSCs due to their compact structure and low porosity. Therefore, it is of high significance to develop UHPCs that have better fire resistance.

Industrial by-products, such as slag and silica fume, have been widely investigated by researchers as replacement of constituents of concrete to achieve environmental benefit as well as better concrete performance. Steel slag is produced under a high temperature of 1,650 °C so that organic, semi-volatile and volatile compounds are removed. Mainly steel slag consists of calcium silicates, ferrites oxides and compounds of iron, magnesium, manganese and alumina. The physical and chemical analyses showed steel slag aggregate is tough, durable, and of relatively high porosity and density [13]. Guo et al. [14] conducted an experimental study to investigate effect of steel slag powder on workability and durability of concrete. They found compared to addition of ground granulated blast furnace slag (GGBFS) alone to concrete, combined admixture of GGBFS and steel slag could achieve lower dry shrinkage and better abrasion resistance. Especially, when activators such as desulphurization, residue sodium sulfate and desulphurization gypsum were integrated, steel slag significantly improved resistance of the concrete to chloride permeation and water permeation and carbonisation depth. Netinger et al. [15] studied steel slag as a coarse aggregate in concrete and concluded steel slag could be an acceptable aggregate of concrete for structural use and posed no risk in terms of corrosion of reinforcement. Qasrawi et al. [16] investigated the effect of steel slag at different replacement ratios

81 of fine aggregate. They proposed use of steel slag in concrete could greatly enhance

82 strength of concrete, especially tensile strength, if appropriate ratio of steel slag to

83 cement was used. Effect of high temperatures on concrete with inclusion of slag has

84 also been studied by several researchers. Yüksel et al. [17] observed that the residual

85 compressive strength of specimens containing blast-furnace slag or coal bottom ash

86 was lower than the reference concrete that did not contain any slag or coal bottom ash.

87 Netinger et al. [18] suggested steel slag as coarse aggregate could improve fire

88 resistance of concrete only if it was combined with a binder that was able to better

89 adapt to slag expansion under high temperature or used as a partial replacement for

90 coarse aggregate. Silica fume is an ultra-fine reactive powder and inclusion of it can

91 improve properties of concrete particularly in compressive strength, bond strength and

92 abrasion resistance due to pozzolanic reactions between silica fume and free calcium

93 hydroxide (CH) in the cement paste [19].

94

95 To date, some efforts have been made to investigate influence of incorporation of slag

96 into UHPCs [20, 21] or other types of concretes [18, 22-24]. However, there are

97 currently very limited researches with respect to the effect of slag on the performance

98 of UHPCs subjected to high temperatures. As discussed above, UHPCs could

99 experience severe explosive spalling and suffer dramatic strength loss after being

100 exposed to elevated temperatures. On the other hand, steel slag, as a potential

101 desirable aggregate of refractory concrete, is still dumped as a land-fill material in a

102 significant amount today [13]. This study is to develop a UHPC with high fire

103 resistance. To improve fire resistance of concrete, steel fibre and polypropylene (PP)

104 fibre are usually incorporated into concrete mixtures [2, 3, 12, 25, 26]. PP fibre is

105 broadly considered beneficial to control explosive spalling since it melts at around

106 160 °C, leaving a network of escaping channels for vapour and thus helping reduce

107 internal vapour pressure [12, 27-30]. However, so far fire resistance of UHPCs

108 remains a problem of concern even though hybrid fibre is used. In the literature,

109 residual compressive strengths of UHPCs are less than 40% of their room-temperature

110 strength after being exposed to high temperatures below 1000 °C [7, 8, 25]. In this

111 study, by using steel slag as fine aggregate and incorporating other industrial

112 by-products and hybrid fibres, a UHPC with 69% relative residual strength after

113 subjected to 1000 °C has been successfully developed.

114

115 **2. Experimental details**

116

117 *2.1. Materials*

118

119 The cement used in all mixtures in this study was ordinary Portland cement (42.5

120 Grade). The silica fume added to the mixtures had a bulk weight of $200\text{-}250\,\text{kg/m}^3$, a

121 specific surface area of $20\text{-}28\,\text{m}^2/\text{g}$, and a particle diameter of $0.1\text{-}0.3\,\mu\text{m}$. Like

122 silica fume, fly ash is a reactive material and has pozzolanic effect in the concrete

123 mixture which helps convert free CH into calcium silicate hydrate (C-S-H) gel, so it

124 was also applied to all specimens. The fly ash had a standard spherical shape, a

125 density of $2.5\times10^3\,\text{kg/m}^3$, a stacking density of $0.7\times10^3\,\text{kg/m}^3$ and a thixotropic

126 index of 8. Sieve analysis was conducted on the two types of fine aggregate, i.e.

127 quartz sand and steel slag, as shown in Fig. 1. The chemical compositions of cement,

128 silica fume, fly ash and steel slag are listed in Table 1. The quartz sand has a density

129 of $2.65\times10^3\,\text{kg/m}^3$, a hardness of 7 and a melting point of 1,650 °C. It needs to be

130 noted no coarse aggregate was used in any specimens, which avoided expansion

5

131 incompatibility between coarse aggregate and surrounding cement paste under

132 elevated temperatures.

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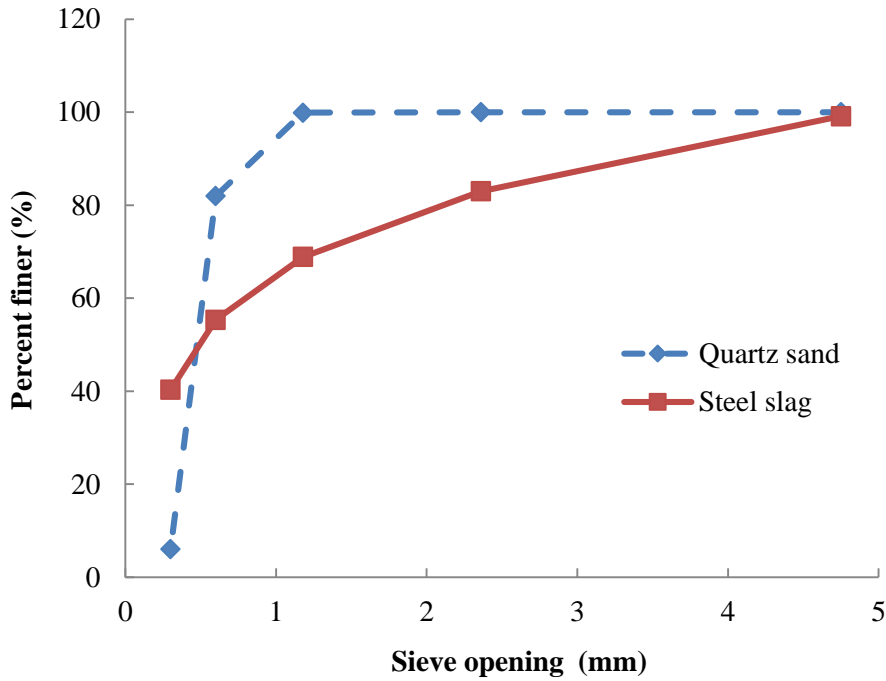
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135 Fig. 1. Sieve analysis of quartz sand and steel slag

136

137 Table 1

138 Chemical compositions of cement, silica fume, fly ash and steel slag (%)

Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Loss of ignition
Cement	21.86	4.25	2.66	63.59	2.19	1.75
Silica fume	93.95	0.5	0.59	1.95	0.27	1.3
Fly ash	52	22	4	12	0.62	<1
Steel slag	17.03	5.64	22.69	43.38	5.98	1.56

139

140 Two types of fibre, i.e. steel fibre and PP fibre, were either or both incorporated into

141 the UHPC mixtures (see Fig. 2). The steel fibre had a round straight shape and the

142   properties of steel fibre and PP fibre are reported in Table 2.

143

(a) Steel fibre   (b) PP fibre

144   Fig. 2. Steel fibre and PP fibre.

145

146   Table 2

147   Properties of steel fibre and PP fibre

	Length (mm)	Diameter (mm)	Density (g/cm^3)	Modulus of elasticity (GPa)	Tensile strength (MPa)	Melting point (°C)
Steel fibre	10	0.12	7.8	-	> 2500	-
PP fibre	10	0.0031	0.91	$\geq$ 3.5	$\geq$ 400	165

148

149   *2.2. Mixture proportioning*

150

151   Totally six mixtures were prepared and they were divided into two series according to

152   different fine aggregates, i.e. quartz sand or steel slag, with details shown in Table 3.

153   The series 1 used quartz sand as fine aggregate, while the series 2 used steel slag.

154   Mixtures in series 1 were divided into four categories according to fibre inclusion, i.e.

155   no fibre, 2 vol.% steel fibre, 2 vol.% PP fibre or combination of 1 vol.% steel fibre

156   and 2 vol.% PP fibre. Mixtures in series 2 were divided into two categories according

157   to fibre inclusion, i.e. no fibre or combination of 1 vol.% steel fibre and 2 vol.% PP

158   fibre. For designation of the six mixtures, the letter before the hyphen denotes the type

159   of fine aggregates, i.e. Q for quartz sand and S for steel slag, while the letter behind the

160   hyphen means the type of fibres, i.e. 0 for no fibre, S for steel fibre, P for PP fibre and

161   SP for hybrid fibre. Water to binder ratio was set at 0.16 and sand to binder ratio at 1.0

162   for all the mixtures. Unit weight for all the mixtures and slump for the mixtures Q-SP,

163   S-0 and S-SP are listed in Table 4. The slump tests were conducted conforming to the

164   Chinese standard GB/T 50080-2016. It can be seen the slump of the Q-SP and S-SP

165   concretes was greatly reduced by inclusion of the hybrid fibre.

166

167   Table 3

168   Mixture proportions of UHPCs (unit: kg/m^3)

Constituents	Series 1				Series 2	
	Q-0	Q-S	Q-P	Q-SP	S-0	S-SP
42.5 cement	850	850	850	850	850	850
Silica fume	137.5	137.5	137.5	137.5	137.5	137.5
Fly ash	112.5	112.5	112.5	112.5	112.5	112.5
Water	176	176	176	176	176	176
Superplasticizer	8	8	8	8	8	8
Quartz sand	1,100	1,100	1,100	1,100	-	-
Steel slag	-	-	-	-	1,100	1,100

Steel fibre	-	156	-	78	-	78
PP fibre	-	-	18.2	18.2	-	18.2

169

170　Table 4

171　Unit weight and slump of UHPC mixtures

	Q-0	Q-S	Q-P	Q-SP	S-0	S-SP
Unit weight (kg/m^3)	2,450	2,610	2,250	2,460	2,590	2,700
Slump (mm)	-	-	-	70	295	75

172

173　*2.3. Specimen preparation and tests*

174

175　The compression tests were carried out for five target temperatures, i.e. 200, 400, 600,

176　800 and 1,000 °C. Under each target temperature, four specimens were prepared and

177　tested allowing for randomness of explosive spalling. There were totally 144 specimens

178　and all specimens had a cubic shape. A side length of 50 mm rather than larger was

179　chosen for all the specimens in order to avoid too frequent occurrence of explosive

180　spalling. To prepare the specimens, a forced mixer with 30 L capacity was used. Firstly,

181　the fine aggregate was fed into the mixer and mixed with a small amount of water for

182　two minutes. Then cement, silica fume and fly ash were added and mixed for five

183　minutes before water and superplasticizer were poured. The mixing process continued

184　for another eight minutes. Finally, fibres were added to the mixture and mixed for five

185　more minutes before the mixture was cast in the steel moulds. 24 hours later, the

186　specimens were demoulded and put into a water tank with thermostatic control, where

187　they were submerged by hot water of constant 90 °C for one day in order to accelerate

188 pozzolanic reaction. Then, they were cured under a standard curing condition for 28

189 days. Before heating, a drying treatment is usually needed to control explosive

190 spalling. Okpala [31] used a mechanically ventilated oven to dry the paste and mortar

191 specimens at 105 ± 1 °C for a duration of 5-6 days. However, after a comparative

192 study of different drying temperatures and facilities, Gallé [32] suggested 105 °C could

193 be too high to prevent microstructure of the specimen from being impacted by oven

194 drying treatment. In this study, the specimens were put into an electric furnace for a

195 drying treatment under a temperature of 90 °C for 24 hours, which proved to be an

196 effective means to control explosive spalling. Since all the six UHPC mixtures were

197 prepared with the identical water content, i.e. 176 kg/m^3, and experienced the same

198 procedure of curing and drying, it was supposed they had the similar initial moisture

199 level.

200

201 The specimens were heated in an electric furnace with a heating capability of 1400 °C.

202 Real fire condition could be simulated as per the heating curve defined in the standards

203 ISO 834 [33] which has an exponential relationship between growing temperature and

204 heating time. However, to make the heating process simple, some researchers such as

205 [34] applied a constant heating rate of 5 °C/min according to the standards ASTM E831

206 [35]. In this study, 4 °C/min was selected as the heating rate according to ASTM E831

207 [35] and 1 and 8 °C/min heating rates were chosen to make a comparison study in

208 subsection 3.2.1. Compact concrete like UHPC is vulnerable to explosive spalling

209 when being heated. To prevent damage to the furnace and ensure safety, silicon boards

210 were mounted on the chamber inner walls of the furnace. Furthermore, a steel cage

211 was presented inside the furnace to contain specimens being heated. At a time, four

212 specimens were heated at a rate of 4 °C/min until reaching the target temperatures and

213   then they remained subjected to the constant target temperatures for another period of

214   time or called holding time to achieve uniformly distributed temperatures throughout

215   the specimen. The holding time, according to Chen et al. [36], has a significant impact

216   on stress-strain behaviour of concretes exposed to high temperatures. To determine an

217   appropriate holding time, a heating simulation with target temperature 1000 °C was

218   conducted using the thermal parameters from the European standard [37]. Fig. 3

219   shows the temperature time-history in the furnace chamber and at the centre of the

220   model. It can be seen after 2 hours holding time (from 245 to 365 min), the

221   temperature at the specimen centre is 999.3 °C, very close to the target temperature.

222   For target temperatures lower than 1000 °C, the temperature gap between the furnace

223   chamber and the centre of the model is smaller, as shown in Fig. 3. Because concrete

224   has higher thermal conductivity and lower specific heat under lower temperatures, it is

225   no doubt the valid holding time for higher target temperature ensures uniform

226   temperature distribution in case of lower target temperatures. Hence, in this study the

227   holding time for all specimens was consistently set at 2 hours to ensure uniformly

228   distributed temperatures, at the same time to avoid impact of this factor. Afterwards,

229   the furnace door was opened to let the specimens cool down to room temperature. It

230   should be noted in this study temperatures were measured by the thermocouple

231   mounted on the internal wall of the furnace unless specified otherwise.

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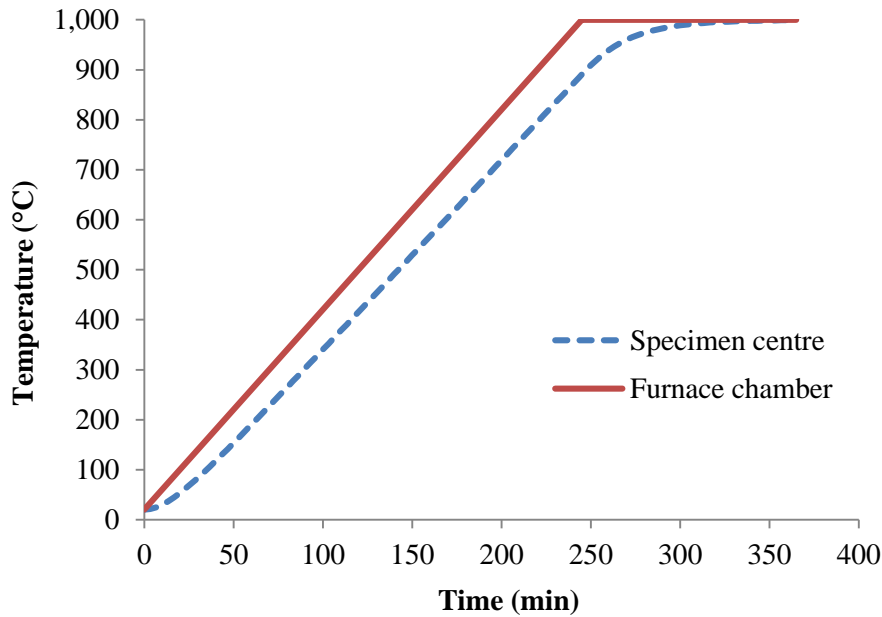
Fig. 3. Comparison of temperature time-history between furnace chamber and specimen centre under target temperature 1000 °C

Compressive tests were implemented on a 1,000 kN capacity electrohydraulic servo pressure testing machine, with the procedure conforming to the Chinese standard JGJ70-2009. The loading rate was controlled at 0.2 mm/min and the loading and displacement data were recorded automatically. To measure the displacement, two displacement gauges were symmetrically mounted on the two sides of the specimen between the loading plates, as illustrated in Fig. 4. In this study, the compressive strength was calculated as the average value of four specimens. Results for all mixtures are reported in Table 5, where f_c denotes compressive strength of the specimens which were not exposed to high temperatures (i.e. control specimens), f_{cT} denotes compressive strength of the specimens which were exposed to high temperatures and f_{cT} / f_c means normalised compressive strength. It should be noted while most of the test results were an average of four specimens, some came from three due to explosive spalling. Due to severe explosion, data for the mixtures Q-0, Q-S and S-0 were not

250   recorded above 200 °C.

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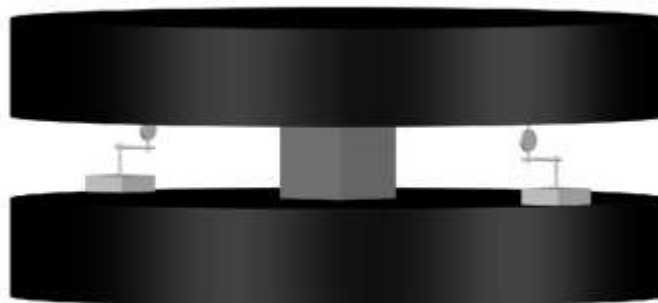
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253   Fig. 4. Sketch of testing setup.

254

255   Table 5

256   Compressive strength under various temperatures

Temperature (°C)		Q-0	Q-S	Q-P	Q-SP	S-0	S-SP
20	f_c (MPa)	112.4	187.5	125.5	162.1	90.0	162.8
200	f_{cT} (MPa)	164.7	204.8	132.5	176.2	191.0	182.5
	f_{cT}/f_c	1.47	1.09	1.06	1.09	2.12	1.12
400	f_{cT} (MPa)	-	-	163.5	230.1	-	215.9
	f_{cT}/f_c	-	-	1.30	1.42	-	1.33
600	f_{cT} (MPa)	-	-	112.3	153.8	-	208.3
	f_{cT}/f_c	-	-	0.89	0.95	-	1.28
800	f_{cT} (MPa)	-	-	38.1	40.0	-	147.6
	f_{cT}/f_c	-	-	0.30	0.25	-	0.91
1,000	f_{cT} (MPa)	-	-	37.5	40.4	-	112.8

| | | f_{cT}/f_c | - | - | 0.30 | 0.25 | - | 0.69 |

3. Results and discussion

3.1. Mass loss

In this paper, mass loss refers to water evaporation and gas escape from UHPCs being heated. The relationship between mass loss and temperature for all the mixtures is depicted in Fig. 5. It can be seen all mixtures had a small mass loss below 200 °C, indicating the UHPC specimens had a very compact structure which hindered vapour from escaping. For mixtures without PP fibres, i.e. the Q-0, Q-S and S-0 concretes, data were not recorded above 200 °C due to the severe explosion. Being heated to 200 °C, both the Q-0 and the Q-S specimens experienced an average mass loss of 1 g or 0.34% in percentage, indicating steel fibre had little influence in mass loss of UHPCs exposed to elevated temperatures below 200 °C. The mass loss of the other three mixtures containing PP fibres increased sharply between 200 °C and 400 °C. It was because the PP fibres melting at 165 °C provided a network of escaping channels for the vapour, while a majority of vapour came from the capillary water and bound water between 200 °C and 400 °C. Research conducted by Tsuchiya and Sumi [38] revealed between temperature range of 360 to 400 °C PP could decompose into various volatiles such as Pentane and Propylene, which was another contribution of the mass loss. Then with temperature growing from 400 °C to 1,000 °C, the mass loss rose slightly and peaked at around 8%. It can be seen from Fig. 5 that the mass loss of the Q-P mixture was a little higher than the Q-SP and the S-SP mixtures. The reason could be the Q-P concrete didn't contain steel fibres and thus suffered severer cracking

14

281   and spalling, which boosted the escaping of vapour and smoke. Another possible

282   reason could be the Q-P concrete as an exception might have more moisture or higher

283   pore volume. The S-SP concrete suffered the smallest mass loss among the three

284   mixtures containing PP fibres, indicating this mixture had more compact structure

285   than the Q-SP concrete, which agreed with the fact that the latter had a lower

286   probability of occurrence of explosive spalling than the former. From 800 °C to

287   1,000 °C, the mass loss of Q-SP and S-SP concretes decreased because they took in

288   moisture from the air during a one-day cooling.

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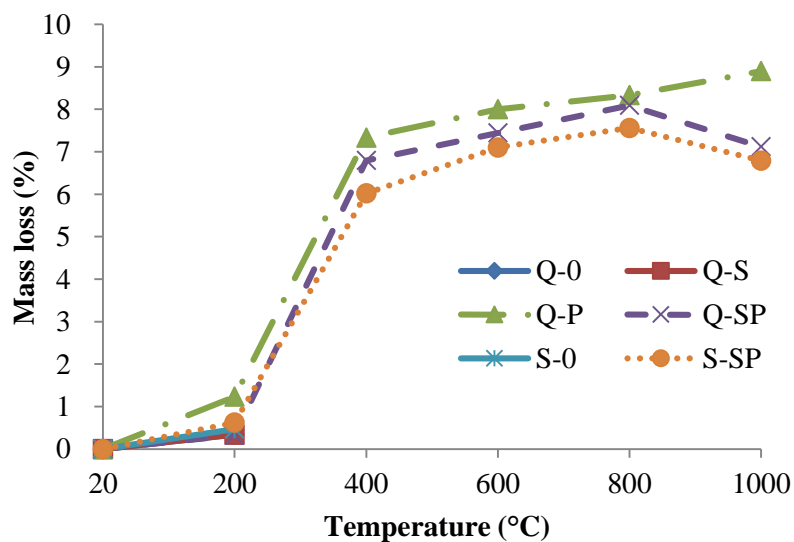
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291   Fig. 5. Mass loss under different temperatures.

292

293   *3.2. Explosive spalling*

294

295   *3.2.1. Effect of heating rate on explosive spalling of the Q-S concrete*

296

297   To investigate the effect of heating rate on explosive spalling of heated UHPCs, three

298   heating rates, i.e. 1, 4 and 8 °C/min, were applied to the Q-S concrete. The target

299   temperature under 1 and 4 °C/min heating rates was set at 400 °C, while under

300   8 °C/min was set at 600 °C.

301

302   The spalling details of the Q-S concrete are reported in Table 6. It was observed under

303   whatever heating rates all the specimens suffered serious explosive spalling, which

304   suggested the heating rate had nothing to do with the probability of explosive spalling

305   in the case of Q-S concrete that was very dense and without PP fibres. However, with

306   the increase of heating rate, the explosive spalling started and ended earlier, both the

307   spalling starting temperature and the spalling ending temperature became higher, and

308   the smoke escaping temperature range became on a higher level. During the heating,

309   there were several intense explosions happening under whatever heating rates, with big

310   burst sounds being heard. Each intense explosion came with the escape of a large

311   amount of vapour and dust, which could be observed outside the furnace. This

312   phenomenon indicated that the accumulation of internal vapour pressure was a big

313   contribution of the explosive spalling.

314

315   Table 6

316   Details of explosive spalling of Q-S concrete at different heating rates

Heating rate (°C/min)	1	4	8
Target temperature (°C)	400	400	600
Probability (%)	100	100	100
Starting time (min)[a]	325	92	35
Ending time (min)[a]	460	113	67
Starting temperature (°C)[b]	323	357	422
Ending temperature (°C)[b]	400	400	563

| Smoke escaping temperature range (°C)[c] | 350-391 | 369-400 | 460-553 |

323

324 Table 7 shows the explosive spalling of the Q-S specimens heated under the three

325 heating rates. It can be seen with higher heating rate and higher target temperature the

326 debris of the specimens tended to be smaller, indicating more intense explosive

327 spalling.

328

329 Table 7

330 Explosive spalling of Q-S concrete at different heating rates

---

Heating rate
(°C/min)

| 1 | | | |

4			
8			

331

3.2.2. Explosive spalling of specimens without PP fibres

333

In this subsection, the explosive spalling of the mixtures without PP fibres, i.e. Q-0, Q-S

and S-0, were compared under the identical target temperature of 400 °C and heating

rate of 4 °C/min. The details of the explosive spalling are listed in Table 8 and Table 9

and the sieving results of debris are shown in Fig. 6. All the mixtures suffered explosive

spalling when being heated to the target temperature as shown in Table 8 and broke into

pieces as shown in Table 9. Accompanying each explosive spalling, it was observed

that some vapour was intensively released. This phenomenon indicated the built-up

vapour pressure was an important contribution to the explosive spalling.

342

Compared with the Q-0 concrete without any fibres, the Q-S concrete reinforced with 2

vol.% steel fibres started exploding at a higher temperature. It was because explosive

spalling would not happen until the internal vapour pressure was greater than the tensile

strength of the concrete which was considerably increased by the addition of steel fibre.

The S-0 concrete which had steel slag as fine aggregate had almost the same spalling

348 starting time but higher starting temperature in comparison with the Q-0 and Q-S

349 concretes which had quartz sand as fine aggregate.

350

351 Table 8

352 Details of explosive spalling of mixtures without PP fibres at heating rate of 4 °C/min

Mixture	Q-0	Q-S	S-0
Target temperature (°C)	400	400	400
Probability (%)	100	100	100
Starting time (min)[a]	86	92	92
Ending time (min)[a]	109	113	147
Starting temperature (°C)[b]	338	357	385
Ending temperature (°C)[b]	400	400	400

353 [a,b] Same as in Table 6.

354

355 Table 9

356 Explosive spalling of mixtures without PP fibres at heating rate of 4 °C/min

Mixture			
Q-0			

| Q-S | | | |
| S-0 | | | |

357

Fig. 6 exhibits sieving results of debris of the three mixtures without PP fibres, which

reflects effect of steel fibre and difference between the two types of aggregate on

explosive spalling. Among the three mixtures, the Q-S concrete had the greatest

proportion in both large and small debris sizes as shown in Fig. 6. The steel fibres

bridged the gaps when the cracks were growing and thus restrained propagation of the

cracks, which helped the Q-S concrete remain more large-size debris than the other two

without steel fibres. On the other hand, the constraint effect of the steel fibres caused

greater vapour pressure, which resulted in more small-size debris after the specimens

exploded. Compared to the Q-0 concrete, the S-0 concrete containing steel slag had a

higher percentage of debris with sizes over 4.5 mm. The reason could be the steel slag

produced under 1,650 °C had relatively high thermal stability and similar chemical

compositions to the cement, which reduced thermal incompatibility between the fine

aggregate and the cement paste.

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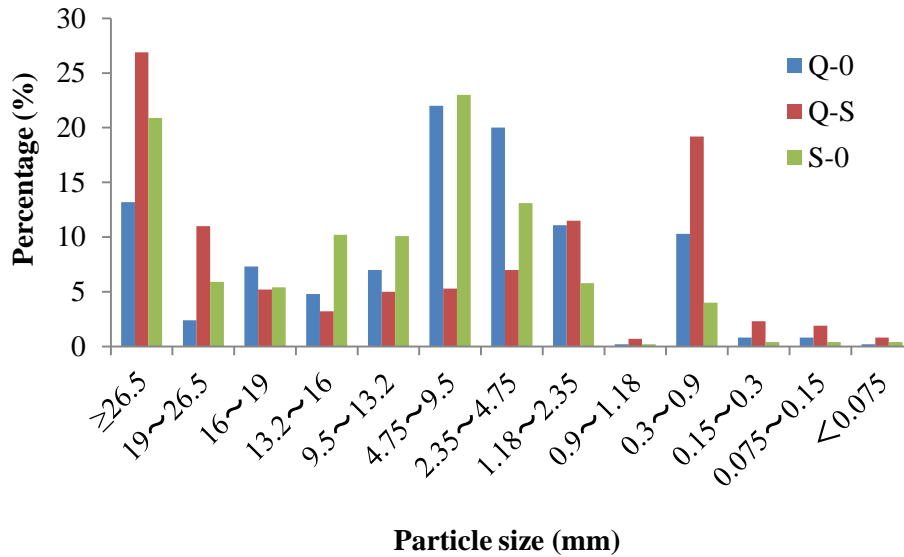
Fig. 6. Sieving results of debris of mixtures without PP fibres.

3.2.3. Explosive spalling of specimens with PP fibres

As discussed above, PP fibre, which melts at about 165 °C, helps release vapour pressure and thus reduces the risk of explosive spalling when concrete is exposed to elevated temperatures. In this subsection, three mixtures containing PP fibres, i.e., the Q-P, Q-SP and S-SP concretes, are compared in terms of explosive spalling. The three concretes were heated to five target temperatures, i.e. 200, 400, 600, 800 and 1,000 °C, with explosive spalling details reported in Table 10. Spalling damage of these specimens is displayed in Table 11, where unheated specimens are also listed in order to make a comparison.

Table 10

Details of explosive spalling of mixtures with PP fibres under different target temperatures

Target tempera-ture (°C)	Spalling probability (%)			Spalling temperature range (°C)		
	Q-P	Q-SP	S-SP	Q-P	Q-SP	S-SP
200	0	0	0	-	-	-
400	0	0	50	-	-	392-400 [a]
600	75	50	50	404-458	447-464 [a]	467-503 [a]
800	75	0	50	409-472	-	391-440 [a]
1,000	100	50	25	413-463	437-457 [a]	464 [a]

389 [a] Only slight spalling was observed.

390

391 From Table 10 and Table 11, it can be seen all the specimens didn't experience

392 explosive spalling at the target temperature of 200 °C. Under the 400 °C target

393 temperature, half of the four S-SP specimens suffered slight spalling but the other two

394 remained intact. With target temperature over 600 °C, all the specimens experienced

395 spalling to some degree, which occurred between 391 °C and 503 °C. However, unlike

396 in the tests of the mixtures without PP fibres, no intensive release of vapour was

397 observed coming with any of the spalling. The spalling could result from thermal stress

398 and incompatibility between the aggregate and the cement paste but not vapour

399 pressure. It can also be seen from Table 10 and Table 11 that the Q-P concrete which

400 had only PP fibres suffered more frequent and severer spalling than the other two

401 reinforced with hybrid fibres. Steel fibre failed to prevent the occurrence of explosion

402 resulting from built-up vapour pressure as discussed in subsection 3.2.2, but it reduced

403 probability and intensity of spalling resulting from thermal stress and incompatibility

404 between the aggregate and the cement paste. When being heated from 20 °C to 1,000 °C,

405 all the specimens experienced a colour change from grey to grey-white and then amber,

406 as shown in Table 11.

407

408   Table 11

409   Explosive spalling of mixtures with PP fibres under different temperatures

Tempera-ture (°C)	Q-P	Q-SP	S-SP
20			
200			
400			
600			
800			
1,000			

410

411   *3.3. Compressive tests*

412

413   *3.3.1. Specimens without PP fibres*

414

415   Because of severe explosive spalling when being heated to 400 °C and above, the

416   specimens without PP fibres were tested only at room temperature and 200 °C. For the

417   compressive tests, failure forms of the three mixtures, i.e. Q-0, Q-S and S-0, are

418 displayed in Table 12. Without addition of any fibres, both the Q-0 and S-0 concretes

419 suffered brittle damage and were crushed up under either temperature. By contrast,

420 the Q-S concrete reinforced with steel fibres improved a lot in ductility and remained

421 its integrity under the compressive loading. The UHPC mixtures had highly compact

422 structure, leading to reduced relative stiffness to the steel loading plate. The internal

423 energy of the loading plate building up during the loading procedure was released

424 suddenly when the specimen yielded, which resulted in the crush of the specimens

425 without steel fibres due to inadequate tensile strength.

426

427 Table 12

428 Failure forms of mixtures with PP fibres under different temperatures

Temperature (°C)	Q-0	Q-S	S-0
20			
200			

429

430 Stress-strain curves for the three mixtures under room temperature and 200 °C are

431 shown in Fig. 7. It can be seen that at 200 °C all the three mixtures enjoyed an

432 improvement of compressive strength in comparison with at room temperature. The

433 reason could be that high-pressure moisture failing to escape due to the tight structure

434 of the UHPC specimen created an enclosed high-temperature and high-pressure curing

435 environment or "internal autoclaving" [39]. This curing environment greatly boosted

436　both cement hydration and pozzolanic reactions due to the presence of active SiO_2

437　from silica fume and fly ash, generating more C-S-H gel. There the C-S-H gel was

438　transformed into xonotlite and tobermorite, which made the internal structure of the

439　specimen even more compact and thus enhanced its compressive strength. As shown

440　in Table 5, when being heated to 200 °C the Q-0 concrete had a 47% increase of

441　compressive strength, while it was only 6% for the Q-P concrete in comparison with

442　their respective original room-temperature strength. It was because PP fibre enhanced

443　the room-temperature strength of the Q-P concrete but its melting at 165 °C made this

444　enhancement invalid. On the other hand, the melting of PP fibres left pores in the

445　concrete, which resulted in more cracks when the concrete was under pressure and

446　reduced moisture pressure due to the release of vapour. These weakened the boost

447　effect resulting from the high-temperature and high-pressure curing environment. From

448　Table 5, it can also be seen the Q-S concrete had an unimpressive 9% improvement of

449　compressive strength compared to 47% for the Q-0 concrete and 112% for the S-0

450　concrete when exposed to 200 °C. The reason is the Q-S concrete reinforced with steel

451　fibres had comparatively high compressive strength at room temperature. Fig. 7 also

452　shows the Q-S concrete had much higher ductility and toughness than the other

453　concretes under either temperature.

454
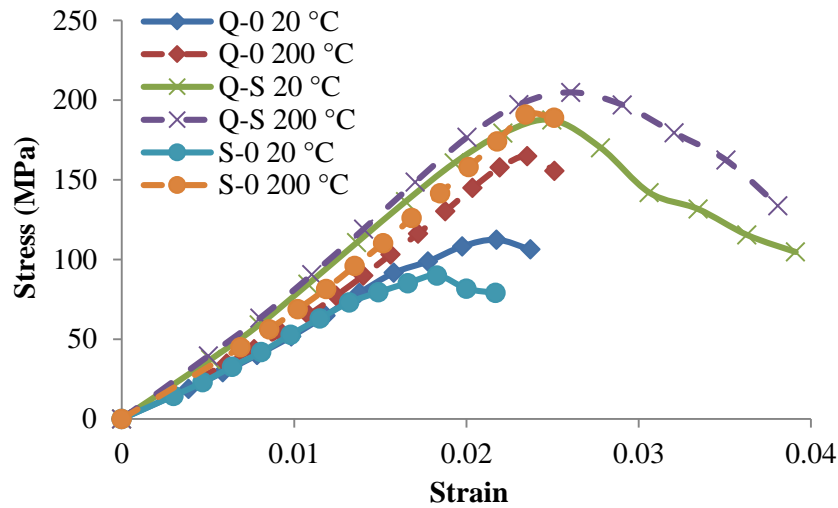
Fig. 7. Stress versus strain curves.

3.3.2. Specimens with PP fibres

Table 13 shows the failure forms of the three mixtures containing PP fibres exposed to temperatures ranging from 20 °C to 1,000 °C. As can be seen, the Q-P concrete suffered a ductile damage at room temperature, but under temperatures 200, 400 and 600 °C its damage was in a brittle manner. It was because the PP fibres improved the ductility of the Q-P concrete under room temperature. However, when it was heated to 200 °C and above, the melted PP fibres resulted in deterioration of ductility of the concrete. Under 800 °C and 1,000 °C, the Q-P concrete exhibited a ductile failure mode again. This was because the concrete had been loosened and softened. Rashad et al. [39] pointed out coarsening of pore-structure can happen when cement paste is heated to 400 °C and above and C-S-H can be dramatically decomposed at 600 °C and above, which may lead to concrete loosening and softening and cohesiveness deterioration of cement paste. Due to the cohesiveness deterioration of the cement paste, a double pyramid was formed during the compressive test on the Q-P concrete subjected to 800 °C and 1,000 °C, as shown in Table 13. Compared to the Q-P concrete, the Q-SP

26

474 and S-SP concretes experienced ductile damage forms under compression throughout

475 all the target temperatures from 20 °C to 1,000 °C. Not only did the addition of hybrid

476 fibres restrain occurrence of severe explosive spalling, but also it greatly improved the

477 ductility of the concrete exposed to high temperatures. Under 1,000 °C, however, the

478 Q-SP and S-SP concretes had the similar failure form to the Q-P concrete. This could be

479 attributed to the loss of strength of steel fibres under high temperatures. According to

480 the European standard [40], under 800 °C and 1,000 °C steel retains only 11% and 4%

481 of its yield strength respectively.

482

483 Table 13

484 Compressive failure forms of mixtures with PP fibres under different temperatures

Temperature (°C)	Q-P	Q-SP	S-SP
20			
200			
400			
600			
800			

1,000	

485

486   The relationships between compressive strength and temperature for the three

487 concretes are illustrated in Fig. 8. With the target temperature ascending from 20 °C to

488 400 °C, all the three concretes made an improvement in compressive strength. From

489 room temperature to 200 °C, the enhancement mechanism is the same as discussed in

490 subsection 3.3.1. From 200 °C to 400 °C, more C-S-H gel was produced and

491 transformed into xonotlite and tobermorite as pozzolanic reactions continued under

492 the high-temperature and high-pressure environment. In addition, evaporation of

493 bound water made the cement paste more compact and harder. Also during this stage,

494 PP fibres melted completely, greatly relieving vapour pressure. All these contributed

495 to an enhancement of compressive strength of the three concretes.

496
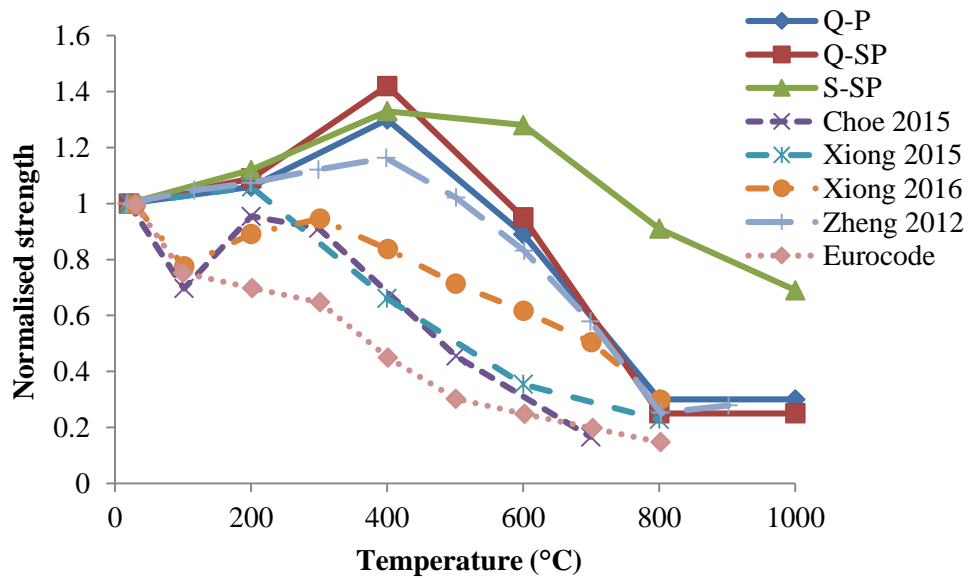
497

498   Fig. 8. Normalised compressive strength versus temperature.

499

500 In contrast, from 400 °C to 800 °C all the three concretes suffered a strength decrease. It

28

can be seen from Fig. 8, the strength loss of the Q-SP concrete was a little steeper than

that of the Q-P concrete, while the S-SP concrete had a much slower deterioration of

strength than the other two. During this stage, decomposition of the CH occurred

between approximately 430 °C and 600 °C, while for the C-S-H it started at around

560 °C and then became significant above 600 °C [39]. A large amount of C-S-H was

transformed into C_3S and β-C_2S and α-SiO_2 was transformed into β-SiO_2, which

resulted in increased porosity in the paste [41]. At 800 °C, the C-S-H was completely

decomposed and the $CaCO_3$ started to decompose, contributing to decreased

compactness of the internal structure and a lot of small cracks on the surfaces of the

specimen. Also, at this stage, the mismatching volume expansion between the cement

paste and the aggregate played a significant role that worsened the mechanical

performance of the concrete [13, 18].

From 800 °C to 1,000 °C, the strength of the Q-P and Q-SP concretes remained almost

the same but that of the S-SP concrete kept falling. However, the S-SP concrete

retained a relatively high residual strength of 112.8 MPa or normalised residual

strength of 69% after being exposed to 1,000 °C, while that for the Q-P and Q-SP

concretes was only 40 MPa or below 30%.

Fig. 8 also shows the results of UHPCs from other researches [4, 7, 8, 25] and high

strength concrete according to the European standard EN 1992-1-2 [37]. It can be

seen over 600 °C the S-SP concrete retained markedly higher normalised strength than

the other concretes. The main reason could be, on one hand, adding steel slag to the

mixture reduced the amount of CH formed during the hydration, which increased the

fire resistance of the mixture exposed to elevated temperatures [42, 43]. On the other

526　hand, steel slag usually has rough surfaces and thus improves properties of interfacial

527　transition zone (ITZ), which could be critical for concrete under high temperatures.

528　Research conducted by Jia [44] revealed NSC exposed to high temperatures above

529　300 °C exhibited different damage modes under compressive loading. The damage

530　occurred along the ITZ or within the paste other than running through the aggregate

531　which was the case for unheated concretes, indicating high temperature could severely

532　degrade properties of ITZ. Ducman and Mladenovič [13] and Netinger et al. [18]

533　suggested a major factor contributing to strength loss of concrete using steel slag as

534　aggregate is the expansion of steel slag in contrast to the shrinkage of cement paste after

535　exposed to high temperatures. However, in this study steel slag was used as fine

536　aggregate and most of it had a size smaller than 2.5 mm, which could mitigate the

537　negative effect of volume mismatching between the steel slag and the cement paste.

538　Besides, as discussed in subsection 3.2, steel slag had excellent thermal stability and

539　similar chemical compositions to cement, which reduced thermal incompatibility

540　between the fine aggregate and the cement paste and could improve residual

541　properties. From Fig. 8, it can also be seen the S-SP concrete was the only one that had

542　its strength under 600 °C higher than its room-temperature strength.

543

544　Fig. 9 shows the SEM analysis of the S-SP concrete after exposure to temperatures from

545　20 °C to 800 °C, using a JEOL JSM-7800F SEM. For the S-SP specimen never

546　subjected to an elevated temperature, it had continuous and compact C-S-H phase and

547　scattered CH crystals. After exposed to 200 °C, the C-S-H phase in the concrete

548　remained continuous and became more compact while the CH crystals became fewer

549　and smaller. However, due to melting of PP fibre and cracking resulting from vapour

550　pressure, the matrix presented a rougher surface. After subjected to 400 °C, the C-S-H

551 phase had even more compact structure and better integrity while the CH phase

552 continued to reduce in quantity and size. Suffering 600 °C, the C-S-H phase lost some

553 continuity and integrity due to decomposition and became less compact. At the same

554 time, the CH phase completely decomposed. At 800 °C, the C-S-H phase did not exist

555 any longer and the matrix was honeycombed, resulting in greatly worsened concrete
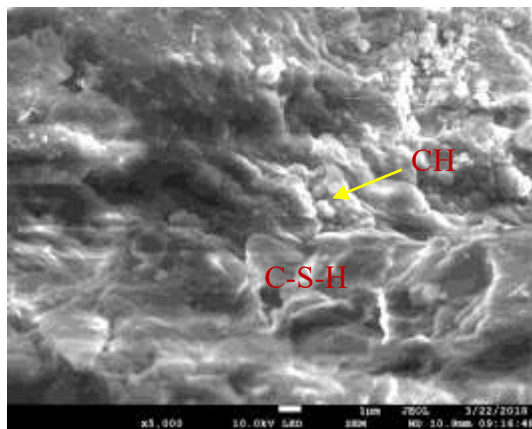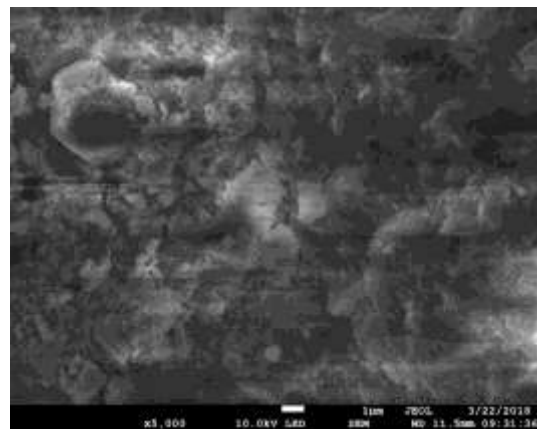
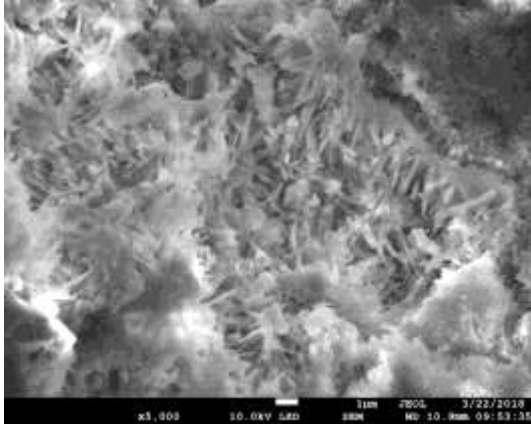556 performance.

557
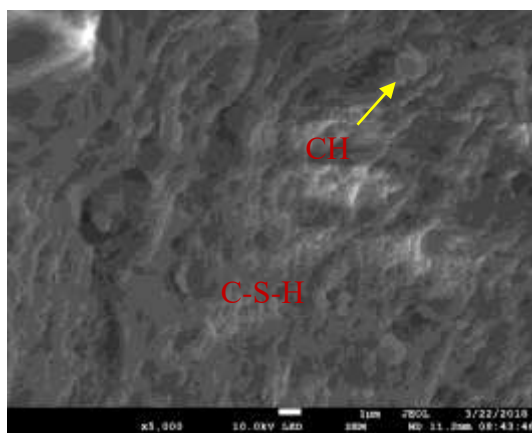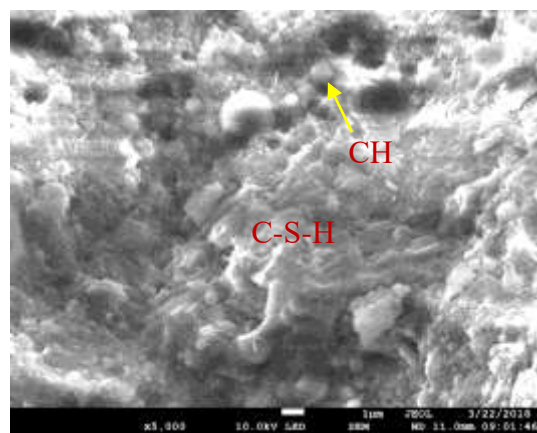
(a) 20 °C
(b) 200 °C

(c) 400 °C
(d) 600 °C

(e) 800 °C

Fig. 9. SEM analysis of S-SP concrete after exposure to different temperatures.

The stress-strain curves of the three concretes under different temperatures from 20 °C to 1,000 °C are depicted in Fig. 10, Fig. 11 and Fig. 12, respectively. It can be seen the stress-strain curves of the Q-P and the Q-SP concretes had the similar varying pattern under different temperatures. For example, both the concretes suffered a significant drop in strength and modulus elasticity when being heated to 600 °C and above and had a flattened stress-strain curve under 800 °C and 1,000 °C. As discussed above, the concretes were loosened and softened when being heated to higher than 800 °C. Nevertheless, due to the addition of steel fibres, the Q-SP concrete exhibited greater ductility and toughness than the Q-P concrete under whatever temperatures. From Fig. 12 it can be seen even under 600 °C the S-SP concrete had strength much higher than its room-temperature strength and relatively high modulus elasticity. Under higher temperatures up to 1,000 °C, the S-SP concrete retained relatively high residual strength and modulus elasticity in comparison with the Q-P and the Q-SP concretes.

574
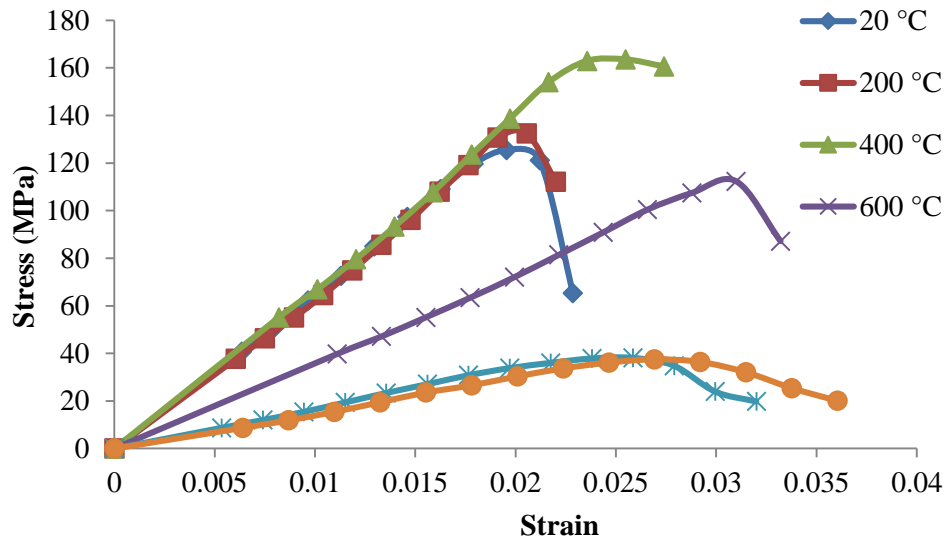
Fig. 10. Stress versus strain curves of Q-P concrete.

576

577

Fig. 11. Stress versus strain curves of Q-SP concrete.

579

Fig. 12. Stress versus strain curves of S-SP concrete.

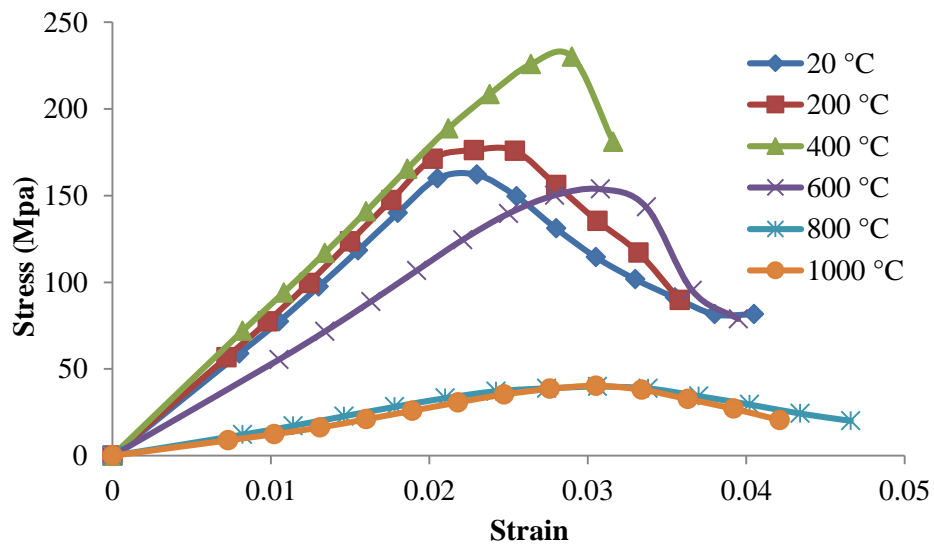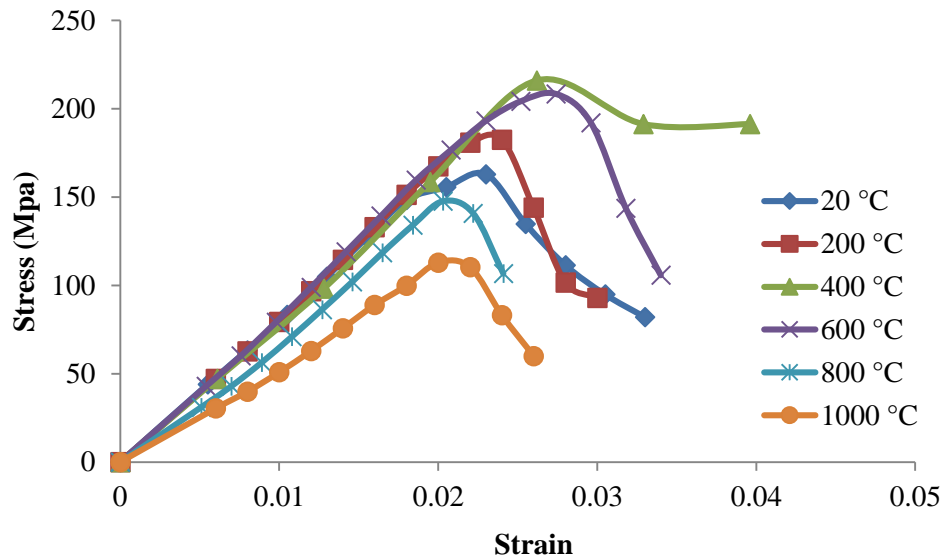Fig. 13 shows relationships between normalised modulus of elasticity and temperature of the three UHPC mixtures as well as light weight concrete from the European standard EN 1992-1-2 [37]. It should be noted modulus of elasticity here was calculated as the slope between two points on the ascending portion of stress-strain curves corresponding to 60% and 40% of peak stress respectively, which was also adopted by Su et al. [45]. The curves for the three UHPCs in Fig. 13 has a similar pattern to those in Fig. 8, though increase of modulus of elasticity was much less impressive than that of strength from 20 °C to 600 °C. It can also be seen the degradation of modulus of elasticity of concrete specified in the European standard EN 1992-1-2 [37] is much severer than the three UHPCs under whatever elevated temperatures.
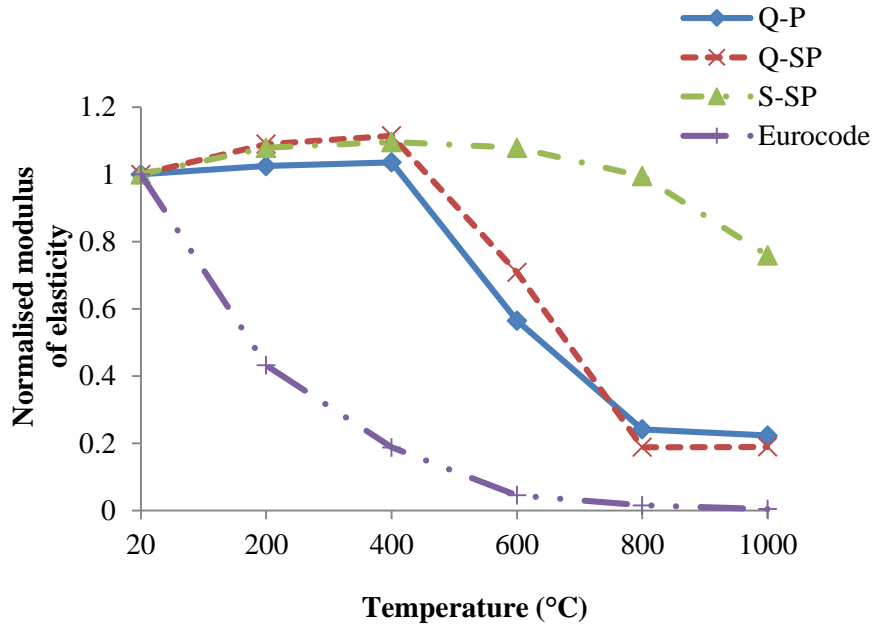
Fig. 13. Normalised modulus of elasticity versus temperature.

4. Conclusions

To develop a UHPC with excellent fire resistance, a total of six UHPC mixtures were designed using quartz sand or steel slag as fine aggregate, with or without steel fibre, PP fibre or hybrid fibre. Special attention was paid to explosive spalling of UHPCs subjected to elevated temperatures since UHPCs are usually of compact structure and thus vulnerable to explosive spalling. Besides, residual compressive strengths and stress-strain relationships of the UHPC mixtures are discussed after they were heated to elevated temperatures up to 1,000 °C. The following conclusions could be drawn from the test results and the above analysis:

1. Most mass loss of the mixtures with PP fibre happened between 200 °C and 400 °C due to water evaporation and PP volatilisation.

2. Heating rate had little effect on explosive spalling occurrence of the Q-S mixture, which had very dense matrix without PP fibres. However, higher heating rate could

35

611   result in more intense explosive spalling.

612   3. Vapour pressure was a significant contribution to explosive spalling. PP fibre was an

613   effective additive to control explosive spalling, though failed to eliminate it

614   completely. Steel fibre could reduce the probability and intensity of spalling

615   resulting from thermal stress and incompatibility between aggregate and cement

616   paste.

617   4. A UHPC mixture with excellent fire-resistance properties was successfully

618   developed. After being exposed to 1,000 °C, it retained 69% of its original

619   compressive strength, much higher than the existing concretes. Reinforced with

620   hybrid fibre, this concrete also greatly decreased the occurrence of explosive

621   spalling.

622   5. Addition of steel slag to UHPC mixtures reduced amount of CH formed during

623   hydration. With rough surface, steel slag could improve ITZ properties of UHPCs

624   exposed to elevated temperatures. Having high thermal stability and similar

625   chemical compositions to cement, steel slag as fine aggregate also relieved thermal

626   incompatibility between aggregate and cement paste. These features of steel slag

627   may significantly improve residual properties of UHPCs using steel slag as fine

628   aggregate.

629

635

636   **References**

637

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