Proceedings of the 6th International Workshop on Concrete Spalling due to Fire Exposure

Sheffield, United Kingdom, 19–20 September 2019

Scientific Editors:
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Prof Ian Burgess

Department
Of
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The University
Of
Sheffield.
Proceedings of the 6th International Workshop on Concrete Spalling due to Fire Exposure

The University of Sheffield
Sheffield, UK
19-20 September, 2019

Dr. Shan-Shan Huang (Chair)
The University of Sheffield

Prof. Ian Burgess
The University of Sheffield

Organizer
Controlling the susceptibility of concrete to spalling during fire exposure is one of the major issues in the design and construction of concrete structures and infrastructure today.

Real fire scenarios indicate that the fire-induced spalling of concrete is capable of producing serious consequences, and is a phenomenon that should be considered when designing buildings for fire resistance. Recent achievements in concrete mix design have led to new types of concrete which, apart from an increased performance at ambient temperature, have also shown an enhanced sensitivity towards fire spalling. However, the fire spalling of concrete is far from being fully understood, and more research is needed to control its likelihood.

The 6th International Workshop on Concrete Spalling due to Fire Exposure concentrates on real-world experiences and observations, practical applications, experimental and numerical advances, as well as structural design. The aim of the workshop is to obtain an overview of the current level of knowledge, and to stimulate discussion between researchers and structural designers, authorities and code-making bodies, in order to promote a more universal level of understanding. The workshop covers topics such as codes and standardisation, the effect of spalling on fire resistance, modelling, high-temperature testing of concrete, spalling tests and protective measures.

The workshop has been arranged in collaboration with the RILEM TC 256-SPF – Spalling of Concrete due to Fire: Testing and Modelling.

We welcome all participants in the 6th International Workshop on Concrete Spalling due to Fire Exposure.
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¹ Université de Pau et des Pays de l’Adour, France
² CSTB, France
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Spalling behaviour of UHPC with modified microstructure due to fire load

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ABSTRACT

Ultra High Performance Concrete (UHPC) is well known for its high compressive strength and its outstanding durability. The main reasons for these properties are a very low water binder ratio and the use of pozzolanic reactive filler (e.g. silica fume) which leads to a dense microstructure with very low permeability. These circumstances makes UHPC very sensitive to fire load and therefore it tends to spall excessively.

In this experimental work, some of the possibilities to modify the microstructure are described and applied to UHPC test specimen: Heat treatment at 105°C and 250°C, artificial air voids, hollow glass microspheres and PP-fibres. All specimens were stored under various conditions for more than half a year to create different moisture contents for the fire tests, which were following the ISO fire curve.

To describe the material properties, air content, slump flow, flexural strength, compressive strength and the pore size distribution (mercury intrusion porosimetry) were determined. The mass changes of the specimens due to treatment and storage are reported, too. During the fire test, the temperature distribution, beginning and duration of spalling were observed. After the fire test the mass of spalled material and its particle size distribution was determined to describe the spalling behaviour additionally.

For some treatment and storage variations, the fire tests showed the expected spalling behaviour of the specimens. However, other variations delivered surprising results.

In the discussion the spalling effects will be related to the treating methods and the changed microstructure and further to the storage conditions and the connected moisture content.

KEYWORD: UHPC, Spalling, Microstructure, Porosity

1 INTRODUCTION

Heat treatment of UHPC is frequently used to increase strength, although the porosity increases and the pore size distribution changes [1], [2]. At a temperature between 230 to 250°C a lot of water is released from amorphous CSH-phases which is a hint of the creation of crystalline Xonotlite that increases strength [3]. Describing the influence of heat treatment of UHPC on the spalling behaviour was one goal of this work.

For some UHPC mixtures PP-fibres were used to prevent spalling. The mode of action of PP-fibres is well described in [4]. These mixtures were also heat treated and the fibres melted during the treatment. Additionally, mixtures with hollow glass microspheres and air-entraining agent were investigated.
2 EXPERIMENTAL PROGRAM

2.1 Mix compositions and components

The mix compositions of all four different mixes are depicted in Table 1. The ratio between cement, silica fume and quartz powder was the same for all compositions. The water cement ratio w/c was 0,28 and the volume ratio water/fines w/fV was 0,45. As a reference (REF) a common UHPC mix was used. As a first variation PP-fibres were added to this reference mix (F-REF).

Table 1: Mixture composition for 1 m³ concrete, air void content took into account

<table>
<thead>
<tr>
<th>Components</th>
<th>REF [kg/m³]</th>
<th>F-REF [kg/m³]</th>
<th>HGM [kg/m³]</th>
<th>AEA [kg/m³]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 52,5 N C₃A free</td>
<td>681,2</td>
<td>681,2</td>
<td>652,3</td>
<td>581,2</td>
</tr>
<tr>
<td>Silica fume (SF)</td>
<td>170,3</td>
<td>170,3</td>
<td>163,1</td>
<td>145,3</td>
</tr>
<tr>
<td>Quartz powder grade 10000</td>
<td>340,6</td>
<td>340,6</td>
<td>-</td>
<td>290,6</td>
</tr>
<tr>
<td>Hollow glass microspheres</td>
<td>-</td>
<td>-</td>
<td>32,6</td>
<td>-</td>
</tr>
<tr>
<td>PP-fibres 6/0,15mm</td>
<td>-</td>
<td>3,00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Quartz sand 0,1-0,5mm</td>
<td>895,7</td>
<td>895,7</td>
<td>901,0</td>
<td>766,7</td>
</tr>
<tr>
<td>Superplasticizer 1 (SP1)</td>
<td>30,7</td>
<td>30,7</td>
<td>26,1</td>
<td>26,2</td>
</tr>
<tr>
<td>Superplasticizer 2 (SP2)</td>
<td>13,6</td>
<td>13,6</td>
<td>13,0</td>
<td>11,6</td>
</tr>
<tr>
<td>Air-entraining agent</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>2,00</td>
</tr>
<tr>
<td>Water incl. liquid part of SP1 and SP2</td>
<td>190,8</td>
<td>190,8</td>
<td>182,6</td>
<td>159,7</td>
</tr>
<tr>
<td>Fresh concrete density</td>
<td>2290</td>
<td>2290</td>
<td>1950</td>
<td>1950</td>
</tr>
<tr>
<td>Air voids in fresh concrete [vol.%]</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>18</td>
</tr>
</tbody>
</table>

For the second variation hollow glass microspheres (HGM), sometimes simply called glass bubbles, were used to replace the quartz powder by volume. Such spheres are sometimes used in building materials that reduce density and thermal conductivity. The properties of the used HGM are depicted in Table 2. The fineness is close to cement. As a third variation an air entraining agent was added (AEA). This type of admixture is usually used to improve frost resistance of concrete and creates pores with a diameter of mainly 300 µm. The amount of hollow glass microspheres and air-entraining agents was chosen in such a way that a comparable concrete density of the two mixtures was achieved.

Table 2: Properties of the used hollow glass microspheres

<table>
<thead>
<tr>
<th>Average density [g/cm³]</th>
<th>Isostatic compressive strength [MPa]</th>
<th>Volumetric particle size distribution [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>D10</td>
<td>D50</td>
</tr>
<tr>
<td>0.22</td>
<td>2.8</td>
<td>20</td>
</tr>
</tbody>
</table>
2.2 Mixing

The concrete was prepared with an Eirich R02 intensive mixer by using the pin-type rotor. All the dry components (except HGM) were mixed for 180 s. The water and all additives were added within the following 30 s. The wet mixing time was 120 s. The tool speed of the mixing tool was 7.9 m/s for the whole mixing process. The mixes with HGM were prepared in the same way, however, the HGM’s were added after wet mixing and mixed another 3 min with the minimum tool speed (0.4 m/s). This was necessary to avoid breaking the glass bubbles during the intensive mixing process.

2.3 Test specimen

Prisms measuring 40 x 40 x 160 mm were used to determine the hardened concrete’s properties. The filled prism formwork was removed with a trowel and compacted on a vibrating table for approx. 30 s. In order to protect the samples from premature drying, they were covered with a polyethylene film.

The dimensions of the unreinforced test specimens for the fire tests were 250 x 300 x 40 mm. Type K thermal couples were installed in the test specimens at different heights in order to record the temperature curves through the test specimens. The thermocouples were arranged in the middle of the specimens starting directly in the fire surface, then in 1 cm, 2 cm and 3 cm depth.

For each mixture two accompanying specimens of the same size like the fire test specimen were prepared to determine the water content.

After about 24 hours, the specimens were demoulded, wrapped in plastic film and stored at room temperature until further treating or changes in storage conditions were conducted.

2.4 Treating and storage

For the fire tests, 20 test specimens were produced for each mixture variation. Ten for the fire tests and ten to determine the water content. Thus, two fire slabs and two water content slabs were available for each type of treating and storage. In addition, three prisms, which were subjected to the same treating and storage conditions, were produced to determine the compressive and flexural tensile strengths for each fire slab. The hardened concrete properties were determined after the fire test of the respective fire slab.

Figure 1 shows the principle of treating and storage.

Figure 1: Principle of treating and storage

In the figure above means:

Mix: The four different mix compositions (REF, F-REF, HGM, AEA).
Climate (C): Storage in a climate chamber at 20°C and 65% rel. humidity.
Water (W): Storage under water at room temperature until fire test.
A250: Heat treating at 250°C hot air in a drying oven. The heating and cooling rate was 0.1 K/min to avoid micro cracking due to a thermally induced stress gradient. The maximum temperature of 250°C was kept for 5 hours. This treating took around 80 hours in total.

A105: Heat treating at 105°C hot air in a drying oven. The heating and cooling rate was 0.1 K/min. The maximum temperature of 105°C was kept up to the day prior to fire testing and these specimens were assumed to be dry.

The abbreviations already used give the test specimens their name. As an example, REF_A250_C_1 means: Reference mix heat treated at 250°C hot air and after stored in the climate chamber until the fire test, specimen nr. 1.

The first test series was carried out at an average concrete age of 245 days and the second one at an average age of 273 days.

2.5 Test set up for fire test

The test furnace was simply made of fire-resistant AlSi bricks and heated with a propane gas burner following the standard ISO-fire curve (Figure 2). The test specimen was placed on the top of this furnace. The heated area was 23x25 cm. The temperature in the furnace was controlled by the use of mantled thermocouples Typ K. A special feature of the furnace was the 45° inclined bottom. Through a flap the spalled material slipped directly into a metal box. The grading curve was later determined from this material in order to describe the spalling behaviour better.

![Test furnace](image)

Figure 2: Test furnace

3 RESULTS

3.1 Mechanical testing

The compressive strength and the flexural strength of all mixes at the age when fire tests were carried out are depicted in Figure 3. Flexural strength was obtained from a 3-point bending test of 6 prisms 40x40x160 mm (3 prisms corresponding to each fire test specimen). Compressive strength was obtained from 6 prisms half the flexural specimen in size of 40x40x40 mm.

The strength values of untreated specimen were in a common range for UHPC. Heat treated specimens always had a higher strength. Heat treated specimens at 105°C had a higher
strength than specimens treated at 250°C, which was unexpected. Probably the very long treating time at 105°C led to these results. No further investigations to clear up this circumstance were carried out for this work. Specimen with HGM and AES had of course a lower strength related to the lower density.

![Figure 3: Strength of all mixtures](image)

### 3.2 Water content

To determine the water content of the fire spalling specimen the two accompanying treated and stored slabs were took as the references. On the day of the fire test the accompanying specimens were put in the drying chamber at 105°C. It was assumed, that the water content was equal to the fire test slabs. UHPC dries very slowly, however, after approximately 200 days the mass changes were negligibly low and the specimens were assumed to be dry. That means, the result for the water content was available 200 days after the fire test. In Table 3 the temporal course of changes in water content of all mixture-treating-storage variations is listed.

It starts from the added water during mixing to the residual water content after the fire test. Depending on the mix composition the mass of a fire test specimen was approximately between 5.8 kg and 6.7 kg and the added water during mixing therefore weighed 450 g to 550 g. This was the mass of water in a test specimen (\( w_{\text{init}} = 100\% \)), with air voids taken into account. The water change due to heat treating \( \Delta w_{\text{heat}} \) was around -45% for REF and F-FREF, and around -55% for HGM and AES, for all specimen treated at 250°C \( \Delta w_{\text{heat}} \) was around -70%. The water change due to storage \( w_{\text{stor}} \) of not heat treated specimen was negligibly low for climate chamber storage and 10 - 17% for water storage. For the specimens heat treated at 250°C and stored in a climate room the adsorption was around 28% whereas the water adsorption of the water stored specimens was almost within the same range as the water loss during heat treating before. Next to consider is the water content before fire tests \( w_{\text{fire}} \). Depending on treating and storage, fire test specimens had more or less water inside than added during mixing. This amount of water can be divided in two parts: Water bound in CSH-phases \( w_{\text{csn}} \) and “free” water \( w_{\text{free}} \).

The amount of free water was obtained by drying the accompanying test specimen at 105°C and it is named \( w_{\text{10s}} \) in Table 3. This is a rough estimation because it is known that drying at 105°C also removes some bound water, at least partially. Taking the density of the concrete into account, the bound water \( w_{\text{csn}} \) was quite similar, except for the mixture variations with AES had less \( w_{\text{csn}} \). Next, the water loss during the fire test \( w_{\text{loss}} \) is reported. It was calculated from the mass of the test specimen before the fire tests compared to the mass after the fire tests, including spalled material (if any) collected in the metal box. The last row of the table
shows the residual water content $w_{res}$ of the specimens after the fire tests. For all the specimens, which did not fail during the fire test and therefore fired for 70 min, this value is very low and they were practically dry. Specimens that failed during the fire test are marked with grey lines in the table.

Table 3: Temporal course of changes in water content

<table>
<thead>
<tr>
<th></th>
<th>$w_{init}$</th>
<th>$\Delta w_{heat}$</th>
<th>$w_{stor}$</th>
<th>$w_{fire}$</th>
<th>$w_{105}$</th>
<th>$w_{free}$</th>
<th>$w_{csh}$</th>
<th>$w_{loss}$</th>
<th>$w_{res}$</th>
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<tbody>
<tr>
<td>REF_C</td>
<td>560 100</td>
<td>0 0</td>
<td>5 1</td>
<td>564 101</td>
<td>3,8</td>
<td>253 45</td>
<td>311 55</td>
<td>-469 8</td>
<td>95 17</td>
</tr>
<tr>
<td>REF_W</td>
<td>553 100</td>
<td>0 0</td>
<td>63 11</td>
<td>616 111</td>
<td>4,6</td>
<td>308 50</td>
<td>308 50</td>
<td>-509 8</td>
<td>107 19</td>
</tr>
<tr>
<td>REF_A105</td>
<td>567 100</td>
<td>-243 -43</td>
<td>0 0</td>
<td>324 57</td>
<td>0,0 (3,7)</td>
<td>0 0</td>
<td>324 100</td>
<td>-69 1</td>
<td>255 45</td>
</tr>
<tr>
<td>REF_A250_C</td>
<td>556 100</td>
<td>-371 -67</td>
<td>150 27</td>
<td>335 60</td>
<td>0,3</td>
<td>18 5</td>
<td>316 95</td>
<td>-231 4</td>
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<td>302 49</td>
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<td>320 69</td>
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<td>4,7</td>
<td>269 58</td>
<td>198 42</td>
<td>-394 7</td>
<td>73 16</td>
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</table>

3.3 Porosity

The porosity was determined by the use of Porotec Pascal 140/440 Mercury Intrusion Porosimeter (MIP). This system can detect pores with a diameter within the range of 3.6 nm up to 116 µm, which is within the range of meso- and macropores. Ultra-macropores and air voids cannot be detected with the used dilatometer. Samples with a volume of 1 cm³ were dried in an evacuated desiccator at room temperature until mass constancy was reached. The total porosity in this pore size range is shown in Figure 4. The mixtures with HGM and AEA showed much higher total porosity than the other mixtures. Probably both, HGM and AEA, contributed partially to pores in this range.

Heat treating increases the total porosity in all mixtures due to de-watering and changes in the CSH-phases.
Furthermore, heat treating at 250°C changes the pore size distribution, too. Figure 5 shows the influence of heat treating at 250°C on pore size distribution and total pore volume of the reference mixture REF.

The total pore volume rose from 6% to 8.7%, which is an increase of 45%. The average pore diameter of the untreated concrete was 8.8 nm and the one of the heat treated concrete 20 nm, which is more than double the size. Heat treating at 105°C did not change porosity that much, although specimens for this work were stored at this temperature for more than half a year.

The total pore volume increased slightly to 6.7% and the average pore diameter to 11 nm. This effect was basically evident in the other mixtures as well, because the basic composition of the paste was always the same. Therefore, the other diagrams are not shown.

### 3.4 Fire tests

The fire tests always lasted until the specimens failed or for 70 min. In Figure 6 the furnace temperatures of all fire test are shown. It can be seen clearly that the furnace temperature of each test was within the limits of the ISO-fire curve. For most of the specimen which failed, a significant temperature decrease occurred during spalling (mainly between minute 10 and 20) due to the release of vapour. To illustrate the temperature measurements in the
specimen the temperature in different depths of REF_A250_C are shown, too. In order not to exceed the length of this article, a further discussion on the temperature course in the test specimens was dispensed with.

Figure 6: Furnace temperature of all fire tests and temperature in different depths of REF_A250_C (for illustration of temperature measurements in the specimens).

Figure 7 shows the temporal spalling behaviour. Both of the specimens of all mixtures showed almost the same behaviour. All mixtures that were heat treated at 250°C survived the fire test, even the reference mix. Almost all of them without any spalling. Only the water stored F-REF_A250_W as well as REF_A250_W and HGM_A250_W showed some spalling.

The mixture REF_C had a lower water content than REF_W but the same porosity. REF_W started spalling earlier and spalling lasted longer until failure.

The mixture REF_A105 treated at 105°C until the fire test showed the longest duration from the beginning to the first spall and then failed very quickly with some big explosions. Although these specimens were dry (no free water) they spalled because of the water released from CSH-phases at higher temperature (late beginning of spalling) and the still tight pore structure which was not changed that much due to heat treating.

The mixture REF_A250_C had a low free water content and did not spall. However, the mixture REF_A250_W with a much higher free water content showed some spalling. Obviously, the higher porosity was not enough to release the vapour quick enough.
The mixtures with PP-fibres F-REF_C, F-REF_W and F-REF_A105 did not spall and the PP-fibres worked properly. The mixtures F-REF_A250_C and F-REF_A250_W showed a similar behaviour to the corresponding REF mixes. The properties of the specimens are also comparable. The PP-fibres already melted during heat treating and not during the fire test. The porosity of F-REF-A250 mixtures is slightly higher (0.4%) than of REF_A250. This could be explained with the voids left by the amount of fibres and some micro cracks formed by expanding PP-fibres before melting. This porosity was obviously not enough to prevent spalling for the water stored F-REF_A250_W, however the mass of the spalled material was very low compared to other mixes (Table 4). The conditions in the specimens during a very slow heat treating process are different from the conditions during a fire test. The working
mechanism of PP-fibres under different conditions might differ and influence the formation of pore structure [5]

The mixtures treated at 105°C with AEA as well as the ones with HGM showed no spalling. All the other AEA and HGM mixtures stored under different conditions spalled and failed like the reference mixtures did. However, spalling behaviour is quite different. The mixtures with HGM and AEA had comparable porosity and water content. However, the mixes with HGM started earlier with spalling and showed a longer spalling duration. The mixtures with AEA started later with spalling and failed very soon after some big explosions. Considering Table 4 and Figure 9 it can be seen, that the spalling behaviour of the HGM mixtures is very similar to the REF mixtures because the hollow space in non broken glass spheres is not accessible as an expansion space for the water vapour produced. For the mixtures AEA the explanation could be similar. The relatively big air voids in relatively long distance to each other entrained by the air-entraining agent were not fully accessible for the vapour through the dense matrix. The start of spalling was only delayed which resulted in stronger explosions.

To describe and illustrate the different spalling behaviour, pictures of some test specimens after the fire test are shown in Figure 8, the masses of spalled material are listed in Table 4 and Figure 9 shows the grading line of the spalled material from the fire tests. This gives, additional to the temporal spalling behaviour, further information.

![Figure 8: Pictures of some test specimen after fire test](image)

When determining the spalled mass, the best possible attempt was made to separate the spalled parts from the remnants of the test specimen. Most specimen failed by cracking and breaking into big parts.

The REF mixtures lost the most material during the fire tests, followed by the HGM mixtures and the AEA mixtures. Comparing the mixing within each group shows that the specimen with a lower water content always lost more material at a higher spalling rate.
Table 4: Mass of spalled material

<table>
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<th>Spalled mass [g]</th>
<th>Spalling rate [g/s]</th>
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<tr>
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<tr>
<td>F-REF_A250_W</td>
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<td>0,1</td>
</tr>
</tbody>
</table>

A higher spalling rate can have two meanings: More small explosions per time unit, producing fine particles or less big explosions per time unit, producing coarse particles.

Figure 9 shows clearly that specimens with higher spalling rate have much coarser grains (D50 between 11,2 and 22 mm) which means lesser, but bigger explosions. The specimens with a lower spalling rate produces much finer spalled material (D50 ~1-2 mm) during a longer time with a high number of small explosion.

Figure 9: Grading line of spalled material

Together with the temporal course of spalling, there is a tendency. The lower the porosity and the higher the water content, the earlier spalling starts, the longer the spalling lasts with an high number of small explosions producing fine graded spalled material, like sand. Even with some higher porosity and some lower water content UHPC could spall. Later, with less, but stronger explosions and a coarse graded spalled material.

4 CONCLUSIONS

Generally heat treating at 250°C can avoid or reduce spalling due to changes in porosity as well as the appropriate use of PP-fibers. However, when UHPC with PP-fibres is supposed to be heat treated at temperatures above the melting point of the PP-fibres, some loss of
protection against spalling could be expected.

The use of hollow glass microspheres and air-entraining agent could not prevent spalling satisfactorily, not even in high doses like used in this work, which led to a strongly reduced strength.

For most of the mixtures the influence of water content and porosity was obvious and led to different spalling behaviour. To describe these differences, sieving the spalled material and calculating the grading line is a simple, but meaningful method.

5 REFERENCES


