1 Some Observations on Testing Conditions 2 of High-Temperature Experiments on Concrete: An Insight 3 from Neutron Tomography

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8 Abstract

9 This communication explores the influence of boundary effects, embedded sensors and
10 crack opening on high-temperature experiments of concrete as revealed by in situ neutron
11 tomography. The hypotheses routinely taken about these experimental aspects in com-
12 mon practice are hereby reassessed in light of the insight given by noninvasive full-field
13 measurements. Notably, we directly assess the heat and moisture insulation techniques and
14 reveal the influence of temperature and gas pressure monitoring on the testing conditions,
15 opening new perspectives toward their improvement.

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16 **Keywords** Concrete · High temperature · Neutron tomography · Boundary effect ·
17 Embedded elements

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18 1 Introduction

19 High-temperature behavior of concrete is an important issue for the structural safety of high-
20 rise buildings, tunnels, nuclear power plants, etc. When these structures are exposed to fire,
21 a phenomenon known as spalling may occur, which consists in the detachment of pieces of
22 concrete from the heated surface. This phenomenon can lead to the premature failure of con-
23 crete structures. For better understanding the physics behind spalling, which is a rather com-
24 plex phenomenon due to the coupled thermo-hydro-mechanical behavior of heated concrete,
25 many laboratory tests have been performed and are documented in the literature. Besides
26 tests on full-scale concrete elements with typical size 3 m (Richter 2004; Boström and Rob-
27 ert 2008), tests on medium-scale specimens with a typical heated surface of 1 m² (Dal Pont
28 and Ehrlicher 2004; Carre et al. 2013; Pereira et al. 2011; Lo Monte and Felicetti 2017) and
29 small-scale specimens (e.g., rectangular prisms or cylinders with edges or diameter/height less
30 than 300 mm) (Kalifa et al. 2000; Dal Pont et al. 2005; Hertz and Sørensen 2005; Van der
31 Heijden et al. 2012; Felicetti and Lo Monte 2013; Tanibe et al. 2014; Toropovs et al. 2015;
32 Lo Monte et al. 2017) have been carried out. In most of these studies, the experimental proce-
33 dure involves embedded elements such as thermocouples and pressure gauges inside concrete
34 specimens to monitor the temperature and gas pressure field. In medium- and small-size tests,
35 which represent only a part of a structural element, some form of heat and moisture insulation
36 is normally applied on the lateral sides of the samples aiming to achieve a uni-dimensional
37 heat and moisture flow. For the moisture insulation, different approaches are routinely used.
38 Self-adhesive aluminum foil was used in Toropovs et al. (2015). Felicetti et al. (2017) tried
39 two different sealing systems: (1) carbon fibers placed on a high-temperature silicon layer pre-
40 viously smeared on the concrete surface and (2) aluminum foil placed on the concrete surface
41 after being both smeared with epoxy resin. The second system turned out to be a better insu-
42 lator according to the mass loss results of a 120 °C drying test in a ventilated oven. Also for
43 the heat insulation, different options can be found in the literature. In Kalifa et al. (2000) and
44 Mindeguia et al. (2010), the lateral sides of the prismatic concrete samples were insulated by
45 porous ceramics blocks. Van der Heijden et al. (2012) insulated their sample with mineral
46 wool. Toropovs et al. (2015) used a 20-mm-thick glass foam layer in their neutron radiogra-
47 phy tests of heated concrete. In Felicetti et al. (2017) and Mugume and Horiguchi (2013), the
48 authors covered their samples with ceramic fiber board.

49 In most of the aforementioned experiments, the validity of the boundary conditions is not
50 directly measured. In this communication, observations from in situ neutron tomography
51 experiments are used to assess the underlying hypotheses. Some of the tests were presented in
52 Dauti et al. (2018a), where the focus was the analysis of the evolution of the moisture profiles,
53 their relation to the temperature profiles, and the influence of the aggregate size on both. Here,
54 we instead focus on some artifacts observed during the experiments, namely the influence
55 of boundary conditions and measurement probes, which can help better appreciate the limit
56 of the hypotheses used when interpreting many experimental results in the literature. These
57 observations rely heavily on the fact that 3D information is available from the tomography
58 tests.

59 **2 Neutron Tomography Experiments on Heated Concrete**

60 In this section, a brief summary of the neutron tomography experiments is reported. More
61 details can be found in Dauti et al. (2018a). Neutron tests show the evolution of moisture
62 within the sample and enable thus much more detailed information than medium and large-
63 scale test. However, neutrons can penetrate concrete only through a few centimeters. This
64 technique is thus only applicable to small samples. These tests were performed using cylin-
65 drical specimens with a diameter of 3 cm, which were cast using commercial plastic con-
66 tainers as mold, alongside with prismatic samples of $40 \times 40 \times 160 \text{ mm}^3$ for mechanical
67 properties. Mechanical properties were determined according to Swiss standard SIA 262-1
68 (similar to the European EN 12390-13 standard). In particular, compressive strength meas-
69 ured at 28 days on 6 samples (halves of initial 3 prisms used to determine flexural strength)
70 resulted in 104.1 MPa. As the primary objective of the experimental study was to inves-
71 tigate the influence of the aggregate size, two different concrete mixes, with maximum
72 aggregate size 8 mm and 4 mm (shown in Table 1), were used. The two mixtures, HPC
73 8 mm and HPC 4 mm, contained alluvial aggregates, like metamorphic rocks, with maxi-
74 mum diameter of 8 mm and 4 mm, respectively. The aggregate volume fraction for both
75 mixtures was of 60% (of the total volume). The specimens were equipped with three ther-
76 mocouples (type K) for temperature measurement at distances 3 mm, 10 mm, and 20 mm
77 from the heated surface (Fig. 1). The two wires of the thermocouples enter the concrete
78 radially from two sides and are welded together at the center. The location of the embedded
79 thermocouples was imposed during concrete casting. In order to end up with a representa-
80 tive concrete heating surface at the top with a minimal amount of air bubbles, the samples
81 were cast upside down with respect to the testing conditions. The thermocouples were thus
82 located toward the bottom during casting. The samples were vibrated on a vibrating table
83 until no more visible air bubbles appeared at the surface. Then, they were sealed in plastic
84 bags and stored in 97% relative humidity and 20 °C. Neutron tomography measurements
85 were performed 28 days after casting.

86 Similarly to Toropovs et al. (2015) and Felicetti et al. (2017), the lateral surface of the
87 sample was covered with self-adhesive aluminum tape, as shown in Fig. 1 (virtually invis-
88 ible to neutrons because of their very low interaction with aluminum) to prevent the vapor
89 from escaping and to obtain a 1D movement of moisture within the heated sample. The
90 aluminum tape used in the tests (3M High Temperature Aluminum Foil Tape 433) has a
91 working temperature up to 316 °C, which is higher than the temperature experienced by
92 the sample during the test ($\sim 310 \text{ }^\circ\text{C}$).

Table 1 Concrete mixtures (kg/
 m^3)

	HPC 4 mm	HPC 8 mm
Cement CEM I 52.5 R	488	488
Silica fume	122	122
Aggregate 0–1 mm	632.8	400
Aggregate 1–4 mm	949.2	600
Aggregate 4–8 mm	0	582
Superplasticizer SIKA 20HE	8.54	8.54
Water	189.1	189.1
w/b total	0.31	0.31

Fig. 1 Specimen equipped with thermocouples and wrapped with adhesive aluminum tape (in testing orientation)

93 The specimen was placed inside a heating cell (Fig. 2). During a test, the specimen was
 94 heated on the top face with a ceramic infrared radiator, which reached 500 °C within 3
 95 minutes and then was kept at constant temperature. The specimens were surrounded with
 96 rockwool, as shown in the drawing in Fig. 2, in order to maximize the uniformity of one-
 97 dimensional heating.

Fig. 2 Drawing of the heating cell

Neutron tomographies were performed at the NeXT (D50) beamline at the Institut Laue-Langevin (ILL) in Grenoble, France. The principle of neutron tomography is analogous to that of X-ray computed tomography. However, in contrast to X-rays, which interact with electrons and whose attenuation thus depends on the atomic number of the atoms they interact with, neutrons interact with the nuclei. The hydrogen atoms of the water molecule highly attenuate neutrons, which makes it possible to detect the evolving moisture content in concrete. In addition, due to the presence of free and chemically bound water, the cement paste has higher neutron attenuation than the aggregates. Thus, the latter can be easily distinguished from the cement matrix. While the sample was being heated, neutron tomographies were performed for measuring in real time the moisture distribution in 3D (Fig. 3). During the heating test, 60 tomographies (3D scans) were taken. Each tomography, which comprised 500 projections, was acquired in only one minute. Such acquisition time was fast enough for following the rapid dehydration process in concrete.

3 Results and Discussion

3.1 Boundary Effects

One of the challenges when performing experiments on heated concrete is generating a uniform, “1D” heat and moisture flow by adopting the appropriate lateral heat and moisture insulation. Despite the applied insulation for heat and moisture, drying at the boundary was observed in all the tested samples. An example of such a phenomenon is shown in the vertical cut of the 3D volume of sample HPC 8 mm given in Fig. 4. Due to the difference in neutron attenuation between the hydrated and dehydrated cement paste, the drying front is evident in the image. It is clear that the front is not uniform and the precautions taken for obtaining a 1D drying front (rockwool and/or aluminum tape) are insufficient.

A hypothetical explanation to this phenomenon could be the fact that because the specimens are cast, a “wall effect” appears, meaning cement paste concentration near the mold. However, this hypothesis is not supported by the observations in Dauti et al. (2018a),

Fig. 3 Setup in the NeXT (D50) beamline at Institut Laue-Langevin (ILL)

Fig. 4 Vertical cut of the 3D volume, captured at min 48 of heating, highlighting the additional lateral drying

where the boundary effect is virtually identical regardless of the aggregate size (4–8 mm maximum diameter).

For investigating further the effect of the self-adhesive aluminum foil, two concrete samples HPC 4 mm, with and without aluminum foil, were tested. As shown in Fig. 5, the same boundary effect is observed in both samples. This shows that the influence of the aluminum tape is minimal.

It is worth noting that, in the previous studies, such boundary effect was either assumed negligible in the analysis (Kalifa et al. 2000; Felicetti et al. 2017; Van der Heijden et al. 2011), or if it was detected, the 2D nature of the tests made it not quantifiable (Toropovs et al. 2015). The 3D measurements of moisture content by neutron tomography permit the observation and quantification of this boundary effect

The direct observation of the boundary effect opens new perspectives for investigating and eventually improving the current experimental practice on lateral insulation for achieving a uni-dimensional drying front. Realistic boundary conditions are not only

Fig. 5 Boundary effect in two samples, with and without aluminum foil

important for reproducing as closely as possible the actual conditions of a concrete element submitted to fire, but it is also critical in quantifying important physical phenomena such as moisture accumulation. The aspects of the interaction of the boundary effect with the quantification of the moisture accumulation are discussed in the following.

In a situation where the drying front is not uniform, 3D measurements of moisture content by neutron tomography become even more relevant, and 1D or 2D measurements (i.e., neutron radiography, NMR, ground penetrating radar) have to be used with precaution. For instance, in neutron radiography, the recorded intensity is related to the neutron attenuation through the thickness of the sample parallel to the neutron beam. A radiography is a projection resulting from the integration of the signal attenuation in the direction of the beam. The 1D profile is then evaluated by averaging the projection perpendicular to the beam (see Toropovs et al. 2015). The boundary effects perpendicular to the neutron beam can be eliminated by considering only an interior region of the radiography. The boundary effect in the beam direction, however, is inherently inextricable in the radiography. This can give misleading results.

As an example, the averaging process as obtained from radiography is compared to the averaging obtained from tomography in Fig. 6. The two processes correspond to two different regions:

- The first region is a disk which excludes the boundaries and represents a region where the front is uniform.
- The second region is a slice in which we exclude the boundaries only in one direction. Such a region would be representative of the one-dimensional moisture profiles usually obtained from neutron radiography experiments.

The thickness of the disk is of 20 pixels, i.e., 4 millimeters. The purpose of a “thick” disk is merely to reduce statistical noise induced by the high speed of the acquisitions. As such, thinner disk is more noisy but also more spatially detailed.

In Fig. 6, the evolution of the average gray value with time for the two regions is compared. Note that what is perceived as gray value in the images is actually directly related to the water content: a high gray value means a high water content. The “gray” images in Figs. 4 and 5 are transformed to color composite images, where blue color means high gray value (high water content). In Fig. 6 the results of the average gray value are normalized to the initial gray value.

A clear difference between the two can be observed. In the disk, an increase in the gray value is seen, while the gray value in the slice doesn’t exhibit such an increase due to the boundary effect, which still exists in one direction. An increase in the core is in fact hidden by a decrease near the boundary. This simple example highlights another aspect of the importance of 3D measurements.

While the example above demonstrates how the boundary effect can cause an underestimation of the moisture accumulation, it was shown in a previous study (Dauti et al. 2018a) that the boundary effect can also cause an overestimation of the moisture accumulation when a neutron imaging artifact such as beam hardening takes place. A simplified model for estimating the influence of beam hardening showed that this artifact, together with lateral drying, makes the moisture accumulation appear more pronounced than it actually is.

Fig. 6 Moisture profiles for two regions: a disk (representing the analysis performed in the current study using the neutron tomography results) and a slice (representing the analysis usually performed using neutron radiography results), both represented schematically in (a). The results given in (b) show how the 1D profiles obtained from neutron radiography (solid line) can be misleading due to the boundary effect which is not visible in the direction of the beam

183 3.2 Thermocouple-Induced Air Bubbles

184 Embedded elements (e.g., thermocouples, pressure sensors) are routinely employed in
 185 experiments for measuring parameters such as temperature or pressure in heated con-
 186 crete, which are useful for understanding the behavior of concrete at high temperature.
 187 Nevertheless, the presence of such elements inside concrete might perturb the sample
 188 and affect the measurement. For instance, in Dal Pont (2004), it is postulated that a
 189 cavity, possibly formed around the head of the pressure gauge, has an influence on the
 190 pressure measurement. Similarly, in Jansson (2008), it is claimed that a cavity might
 191 be formed around a thermocouple, resulting in a plateau in the temperature develop-
 192 ment around 100 °C, which is an experimental anomaly often observed because of the
 193 fact that the cavity becomes water-filled and the thermocouple measures the temperature
 194 inside the cavity.

195 Unlike in the aforementioned works, where possible defects are hypothesized, in the
196 reported neutron experiments this alteration of the samples induced by the presence of
197 thermocouples was observed directly, for the first time. Figure 7 shows a horizontal slice at
198 the position of the thermocouple located 20 mm from the heated surface of a sample with
199 HPC 4 mm concrete mix. Air voids are observed around the thermocouple wire which has
200 a diameter of only 0.25 mm. It appears that these air bubbles rose during casting when
201 vibrating and were trapped in the proximity of the wires.

202 This is an important observation for the experimental community on heated concrete,
203 which indicates that even very thin embedded elements such as a thermocouple can be
204 intrusive and can affect locally the material properties and eventually the measurement that
205 is being performed. Such an artifact can become even more substantial when considering
206 other embedded measurement elements such as pressure sensors, the size of which can
207 range from 2 to 12 mm (Jansson 2013). Similarly to what is observed for the thermocou-
208 ples in Fig. 7, air voids around a pressure sensor could build an escape channel for vapor
209 and potentially affect the pressure measurements, which have been extensively used in lit-
210 erature for validating numerical models. While several hypotheses have been postulated
211 in the literature on the disturbance caused by an embedded element (Jansson 2008, 2013;
212 Mugume and Horiguchi 2011), neutron imaging represents an effective method for having
213 a closer look to such perturbations in order to improve the measurement procedures.

214 Using neutron imaging, it is also possible to accurately determine the exact position of a
215 thermocouple. This useful information becomes particularly relevant when considering the
216 validation of numerical models, as even a small change in the position causes a significant
217 difference in the temperature results as shown in Dauti et al. (2018b).

218 3.3 Influence of a Crack on the Drying Front

219 The concrete samples employed in heating tests may contain preexisting cracks due to a
220 number of reasons including shrinkage, disturbance by the embedded elements, demold-
221 ing, etc. In addition, cracks may form during heating (high temperature gradients, thermal
222 incompatibility aggregate–cement paste, etc). These cracks often go unnoticed since they

Fig. 7 Air voids around a thermocouple in a high-resolution image

are too small to be detected by the naked eye or are located inside the sample. In this section, the detection and the influence that a crack can have on the drying front are discussed.

A heating test in which a crack has been observed to influence the drying front is presented in the following. Note that the experimental procedure followed for this test is the same as the one described in Sect. 2. The sample (HPC 4 mm) was equipped with thermocouples and no crack was observable when looking at the sample with naked eye before the test. Vertical slices of the tomographies, obtained at different times, are shown in Fig. 8. A diagonal crack, initiated at the position of the thermocouple, possibly due to the voids surrounding it, created a diagonal drying path alongside it. The reason may be related to the physical process of dehydration occurring predominantly around a crack, where vapor can escape and the pore pressure is low.

4 Conclusion

Neutron tomography experiments on concrete have revealed important information related to the commonplace experimental procedures employed in the study of the behavior of concrete at high temperature with respect to spalling.

It has been observed that the heat and moisture insulation techniques that have been adopted in this study, as well as in other studies in the literature, are not sufficient, as all the tested samples experience some degree of lateral drying. This yields a non-uniform drying front, which not only affects the representativity of the sample with respect to a concrete element subjected to fire, but is also critical in quantifying important physical phenomena such as moisture accumulation.

Tomography images have shown that air voids can form around thermocouples resulting in a perturbation of the monitored parameters. Such an observation is important for the experimental community, not only in the case of a thermocouple, but also when employing other embedded elements, such as a pressure gauge, as the validation of many state-of-the-art models on heated concrete are based on the pressure measurements.

The 3D information from tomography also gives insight into the influence that a crack, which is not visible with naked eye, can have on the drying front.

It is important to emphasize that the observations presented in this study are based on small-scale experiments ($d = 3$ cm). Such kinds of experiments are crucial for understanding the physical phenomena involved in fire spalling of concrete. In addition, they provide

Fig. 8 Vertical cut at **a** 20 min, **b** 26 min, **c** 30 min and **d** 42 min

important data for validating the numerical models for predicting spalling. Therefore, the authors believe that the findings presented hereby reveal important information for the experimental community on concrete at high temperature and can be employed in the perspective of improving the experimental procedures.

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