

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

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- 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.

¹⁶ Keywords Concrete · High temperature · Neutron tomography · Boundary effect ·

¹⁷ Embedded elements

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18 1 Introduction

High-temperature behavior of concrete is an important issue for the structural safety of high-19 rise buildings, tunnels, nuclear power plants, etc. When these structures are exposed to fire, 20 a phenomenon known as spalling may occur, which consists in the detachment of pieces of 21 concrete from the heated surface. This phenomenon can lead to the premature failure of con-22 crete structures. For better understanding the physics behind spalling, which is a rather com-23 24 plex phenomenon due to the coupled thermo-hydro-mechanical behavior of heated concrete, many laboratory tests have been performed and are documented in the literature. Besides 25 tests on full-scale concrete elements with typical size 3 m (Richter 2004; Boström and Rob-26 ert 2008), tests on medium-scale specimens with a typical heated surface of 1 m² (Dal Pont 27 and Ehrlacher 2004; Carre et al. 2013; Pereira et al. 2011; Lo Monte and Felicetti 2017) and 28 small-scale specimens (e.g., rectangular prisms or cylinders with edges or diameter/height less 29 than 300 mm) (Kalifa et al. 2000; Dal Pont et al. 2005; Hertz and Sørensen 2005; Van der 30 Heijden et al. 2012; Felicetti and Lo Monte 2013; Tanibe et al. 2014; Toropovs et al. 2015; 31 Lo Monte et al. 2017) have been carried out. In most of these studies, the experimental proce-32 dure involves embedded elements such as thermocouples and pressure gauges inside concrete 33 specimens to monitor the temperature and gas pressure field. In medium- and small-size tests, 34 35 which represent only a part of a structural element, some form of heat and moisture insulation is normally applied on the lateral sides of the samples aiming to achieve a uni-dimensional 36 heat and moisture flow. For the moisture insulation, different approaches are routinely used. 37 38 Self-adhesive aluminum foil was used in Toropovs et al. (2015). Felicetti et al. (2017) tried two different sealing systems: (1) carbon fibers placed on a high-temperature silicon layer pre-39 viously smeared on the concrete surface and (2) aluminum foil placed on the concrete surface 40 after being both smeared with epoxy resin. The second system turned out to be a better insu-41 lator according to the mass loss results of a 120 °C drying test in a ventilated oven. Also for 42 the heat insulation, different options can be found in the literature. In Kalifa et al. (2000) and 43 Mindeguia et al. (2010), the lateral sides of the prismatic concrete samples were insulated by 44 porous ceramics blocks. Van der Heijden et al. (2012) insulated their sample with mineral 45 wool. Toropovs et al. (2015) used a 20-mm-thick glass foam layer in their neutron radiogra-46 phy tests of heated concrete. In Felicetti et al. (2017) and Mugume and Horiguchi (2013), the 47 authors covered their samples with ceramic fiber board. 48 In most of the aforementioned experiments, the validity of the boundary conditions is not 49 directly measured. In this communication, observations from in situ neutron tomography 50 experiments are used to assess the underlying hypotheses. Some of the tests were presented in 51

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.

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59 2 Neutron Tomography Experiments on Heated Concrete

In this section, a brief summary of the neutron tomography experiments is reported. More 60 details can be found in Dauti et al. (2018a). Neutron tests show the evolution of moisture 61 within the sample and enable thus much more detailed information than medium and large-62 scale test. However, neutrons can penetrate concrete only through a few centimeters. This 63 technique is thus only applicable to small samples. These tests were performed using cylin-64 drical specimens with a diameter of 3 cm, which were cast using commercial plastic con-65 tainers as mold, alongside with prismatic samples of $40 \times 40 \times 160$ mm³ for mechanical 66 properties. Mechanical properties were determined according to Swiss standard SIA 262-1 67 (similar to the European EN 12390-13 standard). In particular, compressive strength meas-68 ured at 28 days on 6 samples (halves of initial 3 prisms used to determine flexural strength) 69 resulted in 104.1 MPa. As the primary objective of the experimental study was to inves-70 tigate the influence of the aggregate size, two different concrete mixes, with maximum 71 aggregate size 8 mm and 4 mm (shown in Table 1), were used. The two mixtures, HPC 72 8 mm and HPC 4 mm, contained alluvial aggregates, like metamorphic rocks, with maxi-73 mum diameter of 8 mm and 4 mm, respectively. The aggregate volume fraction for both 74 mixtures was of 60% (of the *total* volume). The specimens were equipped with three ther-75 mocouples (type K) for temperature measurement at distances 3 mm, 10 mm, and 20 mm 76 from the heated surface (Fig. 1). The two wires of the thermocouples enter the concrete 77 radially from two sides and are welded together at the center. The location of the embedded 78 79 thermocouples was imposed during concrete casting. In order to end up with a representative concrete heating surface at the top with a minimal amount of air bubbles, the samples 80 were cast upside down with respect to the testing conditions. The thermocouples were thus 81 located toward the bottom during casting. The samples were vibrated on a vibrating table 82 until no more visible air bubbles appeared at the surface. Then, they were sealed in plastic 83 bags and stored in 97% relative humidity and 20 °C. Neutron tomography measurements 84 were performed 28 days after casting. 85

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

	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

 Table 1
 Concrete mixtures (kg/m³)

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



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



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Neutron tomographies were performed at the NeXT (D50) beamline at the Institut

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Laue-Langevin (ILL) in Grenoble, France. The principle of neutron tomography is analo-99 gous to that of X-ray computed tomography. However, in contrast to X-rays, which interact 100 with electrons and whose attenuation thus depends on the atomic number of the atoms they 101 interact with, neutrons interact with the nuclei. The hydrogen atoms of the water molecule 102 highly attenuate neutrons, which makes it possible to detect the evolving moisture con-103 tent in concrete. In addition, due to the presence of free and chemically bound water, the 104 cement paste has higher neutron attenuation than the aggregates. Thus, the latter can be 105 easily distinguished from the cement matrix. While the sample was being heated, neutron 106 tomographies were performed for measuring in real time the moisture distribution in 3D 107 (Fig. 3). During the heating test, 60 tomographies (3D scans) were taken. Each tomogra-108 phy, which comprised 500 projections, was acquired in only one minute. Such acquisition 109 time was fast enough for following the rapid dehydration process in concrete. 110

111 3 Results and Discussion

112 3.1 Boundary Effects

One of the challenges when performing experiments on heated concrete is generating a 113 uniform, "1D" heat and moisture flow by adopting the appropriate lateral heat and moisture 114 insulation. Despite the applied insulation for heat and moisture, drying at the boundary 115 was observed in all the tested samples. An example of such a phenomenon is shown in the 116 vertical cut of the 3D volume of sample HPC 8 mm given in Fig. 4. Due to the difference 117 in neutron attenuation between the hydrated and dehydrated cement paste, the drying front 118 is evident in the image. It is clear that the front is not uniform and the precautions taken for 119 obtaining a 1D drying front (rockwool and/or aluminum tape) are insufficient. 120

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)



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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



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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 143 content by neutron tomography become even more relevant, and 1D or 2D measure-144 ments (i.e., neutron radiography, NMR, ground penetrating radar) have to be used with 145 precaution. For instance, in neutron radiography, the recorded intensity is related to the 146 neutron attenuation through the thickness of the sample parallel to the neutron beam. 147 A radiography is a projection resulting from the integration of the signal attenuation in 148 the direction of the beam. The 1D profile is then evaluated by averaging the projection 149 perpendicular to the beam (see Toropovs et al. 2015). The boundary effects perpen-150 dicular to the neutron beam can be eliminated by considering only an interior region 151 of the radiography. The boundary effect in the beam direction, however, is inherently 152 inextricable in the radiography. This can give misleading results. 153

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

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

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

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

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

218 3.3 Influence of a Crack on the Drying Front

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



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 pre-225 sented in the following. Note that the experimental procedure followed for this test is the 226 same as the one described in Sect. 2. The sample (HPC 4 mm) was equipped with thermo-227 couples and no crack was observable when looking at the sample with naked eye before the 228 test. Vertical slices of the tomographies, obtained at different times, are shown in Fig. 8. A 229 diagonal crack, initiated at the position of the thermocouple, possibly due to the voids sur-230 rounding it, created a diagonal drying path alongside it. The reason may be related to the 231 physical process of dehydration occurring predominantly around a crack, where vapor can 232 escape and the pore pressure is low. 233

234 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-theart 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

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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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