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Analysis of moisture migration in concrete at high temperature through *in-situ* neutron tomography

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ABSTRACT

Spalling, which is a phenomenon encountered when high-performance concrete is exposed to high temperature, can lead to large economical damage and can be a major safety hazard. Moisture distribution in concrete during exposure to fire is of paramount importance for understanding the complex mechanism of this phenomenon. To capture in its fullness this mechanism, it is crucial to account for the heterogeneous nature of concrete.

In this paper, the first 3D analysis of moisture distribution in concrete at high temperature through *in-situ* neutron tomography is presented. The world-leading flux at the Institute Laue Langevin in Grenoble, France allowed capturing one 3D scan per minute, which is sufficient to follow the fast dehydration process. The paper describes the experimental setup with the heating system and discusses in detail the framework of the neutron tomography test. Quantitative analysis showing the effect of the aggregate size on the moisture distribution is presented.

1. Introduction

High-performance concrete is a widely used material in construction industry and is prone to spalling when exposed to fire. Spalling in tunnels, parking areas, nuclear plants *etc.* during large fires often causes the failure of structural elements and can be a major safety hazard and induce grave economical damage. Several preventive methods [1-3] against concrete spalling have been devised and implemented. However, due to its complexity and a lack of direct observation, the physics behind this phenomenon is not yet fully understood.

Different explanations for spalling have been presented in the past. The two principal mechanisms more broadly accepted are the differential thermal gradients [4] and the pore pressure build-up [5]. The latter is related to the evolution of the moisture content in concrete due to phase changes such as dehydration, evaporation, condensation and mass transfer caused by pressure gradients. A combination of these physical processes may result in the formation of a moisture clog that leads to pore pressure build up to which spalling itself is attributed. Experiments measuring temperature and gas pressure [6-11] have been performed to investigate the processes underlying this theory. Nonetheless, the pressures measured in these studies spread over a large range of values (0.3 –3.5 MPa) for comparable types of concrete. This discrepancy can be ascribed to the disparate types of pressure sensors used for the measurements [12,13] as well as to the intrusive nature of experimental procedures themselves.

Other studies attempted therefore the investigation of moisture distribution in heated concrete following alternative approaches, notably employing nuclear magnetic resonance [14,15] and neutron radiography [16,17]. One dimensional moisture profiles were obtained in these experiments, albeit none of them has been able to track the moisture distribution in three dimensions. Spalling is an intrinsically three-dimensional process, since it locally depends on the heterogeneity of concrete. As a consequence, one dimensional moisture profiles are not sufficient for investigating the role of aggregates in the moisture distribution.

To capture in its fullness the complex mechanisms involved in spalling, it is crucial to account for the heterogeneous nature of concrete. The main effect of this heterogeneity is perhaps the thermal incompatibility between the aggregates and the cement paste [18,19]. During hardening of concrete at constant temperature, cracks are formed in the cement paste by autogenous shrinkage [20]. The size distribution of aggregates has been observed to influence the shrinkage

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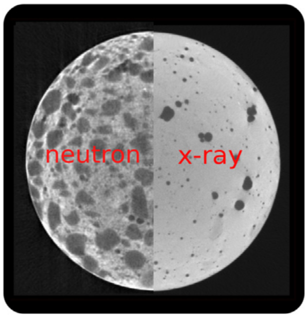

Fig. 1. Joint halves of two horizontal slices of neutron and X-ray tomographies of the same cylindrical sample of concrete (70 mm in diameter) acquired in comparable conditions (~ 100 μm resolutions, polychromatic beams).
Source: From https://next-grenoble.fr/.

cracking [21] and the drying rate of concrete [22]. However, the corresponding studies at high temperature are missing. Measurements of local moisture content in concrete exposed to high temperatures are pivotal for establishing the role of the micro-structure (*i.e.* the grain-scale) in the fire response of concrete.

Neutron tomography is an innovative technique which allows access to the local moisture distribution. The principle of neutron imaging is analogous to that of X-ray imaging, in that both study the attenuation of an incoming radiation (of neutrons in the former and of photons in the latter) and relate it to the local structure of the material studied. X-ray radiation interacts with the outer electrons shells of the atoms it encounters and its attenuation is proportional to the atomic number (and their density). Neutrons interact instead with the atoms' nuclei, leading to counter-intuitive behaviors such as the fact that hydrogen absorbs an order of magnitude more than aluminium or titanium. The particularly high attenuation of hydrogen atoms is especially convenient to study the moisture content (and, therefore, any drying fronts) in concrete. This is highlighted in Fig. 1 where horizontal slices of X-ray and neutron tomographies are compared. X-rays are ideal to detect pores in concrete (because of their low density), but cannot distinguish mortar and the quartz grains used as aggregates, because of their comparable density and atomic number. Conversely, the quartz aggregates are almost invisible to neutrons, which makes their distinction from the mortar straightforward but hinders their differentiation from the pores. This allows investigating the evolution of the moisture content in the cement paste.

This paper focuses specifically on the moisture migration in concrete exposed to high temperatures, which is analyzed for the first time in 3D through *in-situ* neutron tomographies acquired at the Institute Laue Langevin (ILL) in Grenoble, France, which currently has the highest neutron flux in the world [23]. A new imaging instrument named *NeXT* (https://next-grenoble.fr/) has therein recently been developed in collaboration with the Université Grenoble-Alpes. Thanks to its unique flux it has been possible to acquire high-quality tomograms in 1 min, which enables the tracking of the relatively fast process of dehydration at high temperatures, at a resolution sufficient to capture key local heterogeneities.

In Section 2, the experimental setup and the heating system developed for this experimental campaign are detailed, together with the arrangement of the neutron tomography setup in the beamline. The image reconstruction technique employed is also presented therein. Section 3 focuses on the real-time measurements of temperature and moisture distribution. The effect of the aggregates on the moisture distribution is discussed by comparing the drying front in samples with different aggregate sizes. The acquired tomograms are then analyzed in order to quantitatively identify the different phases (notably the aggregates, and the hydrated and dehydrated cement paste). Specifically, the observed evolution of the moisture content in the cement paste is quantified and related to the temperature field. Eventually, the water accumulation behind the drying front, a process pivotal to the pore pressure build-up-driven spalling theory [5], is investigated and quantified.

2. Materials and methods

This section presents the details of the experimental campaign, starting from the preparation of the concrete samples (Section 2.1) to the design of the dedicated testing equipment (Section 2.2). Finally Section 2.3 presents the neutron tomography setup at NeXT and details the passages necessary to reconstruct a 3D image of the sample starting from the acquired radiograms.

2.1. Specimens

As usual for tomography, cylindrical specimens were used. For the diameter, two criteria were considered. In order to investigate the effect of aggregates, the diameter should be large enough to enclose a representative number of aggregates. On the other hand, there is a maximum thickness that can be penetrated by neutrons. Pretests showed that a diameter of 3 to 5 cm yields a good contrast. Although the heating setup has been designed for specimens of up to 5 cm diameter, most tests were performed with 3 cm specimens. Commercial plastic containers were used for the molds (Fig. 2). In some specimens, three thermocouples type K were embedded for temperature measurement at distances 3 mm, 10 mm, and 20 mm from the heated surface (Fig. 3). The two wires of the thermocouples enter the concrete radially from two sides and are welded together at the center. The location of the embedded thermocouples, especially the exact position of the welding point, was determined afterwards from the tomograms. In order to end up with a representative concrete heating surface at the top with a minimal amount of air bubbles, the samples were cast upside down. The thermocouples were thus located at the bottom during casting. To remove any entrapped air, the samples were vibrated on a vibrating table. The samples were sealed in plastic containers which were stored in 97% RH and 20 ° C. In order to remove the surface gloss created by the plastic molds, the heated surface was ground to produce a flat and matt finish, which improves the heat absorption from radiation. To prevent the vapor from escaping and to obtain a 1D movement of moisture within the heated sample, the lateral surface of the samples was covered with self-adhesive aluminium tape (virtually invisible to neutrons because of their very low interaction with aluminium) before the test as shown in Fig. 3. The aluminium 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 maximum temperature experienced by the sample (~ 310 ° C). As one of the objectives was to see the influence of aggregate size, two mixes with

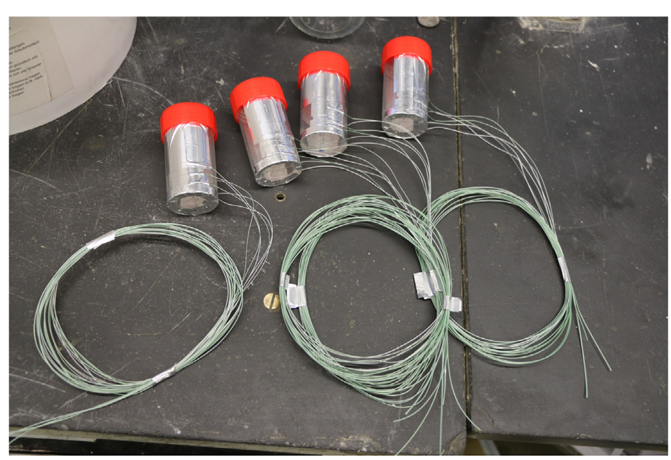

Fig. 2. Molds with thermo-couple wires.