



# Real-time measurements of temperature, pressure and moisture profiles in High-Performance Concrete exposed to high temperatures during neutron radiography imaging



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## ABSTRACT

High-Performance Concrete (HPC) is particularly prone to explosive spalling when exposed to high temperature. Although the exact causes that lead to spalling are still being debated, moisture transport during heating plays an important role in all proposed mechanisms. In this study, slabs made of high-performance, low water-to-binder ratio mortars with addition of superabsorbent polymers (SAP) and polypropylene fibers (PP) were heated from one side on a temperature-controlled plate up to 550 °C. A combination of measurements was performed simultaneously on the same sample: moisture profiles via neutron radiography, temperature profiles with embedded thermocouples and pore pressure evolution with embedded pressure sensors. Spalling occurred in the sample with SAP, where sharp profiles of moisture and temperature were observed. No spalling occurred when PP-fibers were introduced in addition to SAP. The experimental procedure described here is essential for developing and verifying numerical models and studying measures against fire spalling risk in HPC.

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## 1. Introduction

### 1.1. Spalling theories and preventive measures

High-Performance Concrete (HPC) is a modern building material with superior properties compared to traditional concrete [1]. HPC is characterized by high compressive and tensile strengths, high modulus of elasticity, low permeability, high resistance to abrasion and good durability. By reducing the construction dead weight, fewer raw materials are consumed and slender and elegant constructions with lower embodied energy become possible. HPC is used in high-rise buildings, long span bridges, thin walled (especially prefabricated and pre-stressed) elements for building façades, prefabricated columns, elements for tunnels, etc.

A major limitation to the widespread use of HPC comes from its sensitivity to explosive spalling when exposed to fire [2,3]. Spalling reduces the concrete cover of reinforcement and may lead to premature failure of reinforced concrete members. The first theories about spalling

mechanisms go back to the 1960's ([4,5], see also [6]). According to the *pressure build-up theory* [4], water accumulates behind the drying front because vapor produced at the drying front migrates towards the colder inner region, where it condenses. The condensed vapor reduces the gas permeability and may cause liquid water saturation of the pores, referred to as the *moisture clog* [2,7]. At the same time, the high rate of vaporization on the hot side of the moisture clog, together with the thermal dilation of vapor and air due to heating, induces gas pressure build-up in the pores. This process may lead to spalling if the solid pressure acting on the concrete stiff skeleton due to the gas pressure exceeds the tensile strength of the concrete (which at high temperatures is reduced by cracking and dehydration) [8].

Another possible mechanism of spalling is described by the *thermal stresses theory* [5]. Since the temperature at the moisture clog is close to 100 °C and the surface temperature increases rapidly while the depth of the dry layer is still small, a steep thermal gradient develops between the heated surface and the moisture clog, which induces high thermal stresses. According to Saito [5], spalling is due to compression failure near the heated surface. Based on a detailed analysis of pore pressure and thermal stresses, Sertmehmetoglu [4] proposed that the compressive stresses near the heated surface result in tensile cracks parallel to the surface. The gas pressure developing in these cracks, moreover,

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induces explosive spalling. Failure is further eased by stress concentration at the crack tips and by buckling due to compression.

It needs to be underlined that the mechanisms proposed by the two theories (pressure build-up and thermal stress) are strongly coupled and both dependent upon the moisture distribution (and upon the formation of the moisture clog) during fire.

A number of key parameters influencing spalling have been singled out and extensively studied [6,9,10]. However, despite the large body of research, predicting the occurrence of spalling has proven to be an elusive task and current explanations of spalling behavior are mostly empirical and qualitative. It is commonly accepted, however, that high moisture content favors the formation of the moisture clog, slowing down the drying front and increasing the temperature gradient [6]. High heating rate also increases the gradient, while external loads or confinement contribute to increase of the stress [6].

Focusing in particular on the moisture clog, the risk of fire spalling increases when the internal relative humidity (RH) and the water saturation of pores are high, since a higher amount of moisture in the pores may lead to faster clogging. On the other hand, maintaining high RH in HPC is beneficial for reducing self-desiccation shrinkage and early-age cracks. Due to its low water-to-binder ratio (w/b), HPC can experience a considerable decrease of internal RH when the pores are partially emptied of water by the hydration process [11]. As a result, excessive self-desiccation shrinkage may lead both to macroscopic cracking in concrete members and to microcracking within the cementitious matrix. An efficient method for reducing self-desiccation shrinkage and at the same time promoting cement hydration is internal curing by means of superabsorbent polymers (SAP) [12]. The method is based on adding to the mixture dry SAP, that absorb water upon mixing and form small (100–200  $\mu\text{m}$  across) water reservoirs uniformly distributed in the matrix [12]. When the hydration process binds mixing water and empties the capillary pores, water migrates from these water reservoirs and allows maintaining a high internal RH in the concrete, thereby reducing or eliminating self-desiccation shrinkage [12].

An effective method to reduce concrete spalling sensitivity is to add polypropylene fibers (PP-fibers), e.g., [9,13,14], which favor the pore pressure release and limit the formation of the moisture clog [14], thanks to an increase of concrete permeability caused by their melting at about 160–170 °C. PP-fibers have, however, several drawbacks (cost, reduced workability) and the exact mechanisms by which they reduce the probability of fire spalling have not yet been fully understood [14–17]. The combination of PP-fibers and SAP has been recently shown to be beneficial in reducing the risk of fire spalling [18], likely thanks to better connectivity among the fibers enabled by SAP voids, thereby providing better protection against spalling at lower fiber content [18].

Because of the two contradictory premises, namely the higher internal RH needed for reducing shrinkage and cracking, and, on the other hand, the increased risk of fire spalling, studying HPC with SAP addition and relatively high moisture content during heating is of practical relevance and has been the main motivation for this study. Furthermore, the main goal of the present paper is to propose and illustrate with some examples an experimental procedure designed for casting light on the mechanisms behind spalling and provide valuable input for both numerical approaches and mixture design.

## 1.2. Study of mechanisms underlying spalling

To understand the complex mechanisms underlying fire spalling, it is paramount to follow the moisture distribution in concrete during exposure to high temperature, quantitatively and non-destructively, in real time [19]. An effective method to measure water distribution in concrete is neutron radiography [20]. In concrete, aggregates and anhydrous cement weakly interact with neutrons, while hydration products and water-filled capillary pores have the largest interaction (mainly neutron scattering), leading to strong attenuation of an incident neutron beam. The application of neutron radiography to study water

transport in cementitious materials subjected to high temperatures was already suggested in [21]; this idea, however, has been followed only recently [22,23]. Weber et al. [22] performed neutron radiography on small mortar slabs (dimensions  $80 \times 80 \times 10 \text{ mm}^3$ ) standing on a heating element and heated up to 600 °C in 15 min. In order to obtain 1-D temperature and moisture fields, the specimens were insulated against moisture and heat loss using aluminum foil and glass foam, except from the bottom and top edges. Temperature profiles were measured via embedded thermocouples and quantitative moisture profiles were obtained by means of neutron radiography [22].

As concerns pore pressure, several authors have directly performed measurements in concrete specimens subjected to thermal transients (see for instance [2,3,13,24]). This was generally performed by embedding thin stainless steel pipes provided with external electronic sensors. Possible alternatives [25] come from the adoption of a porous sensing head (to measure the mean pressure of larger paste volumes) and filling the pipe with thermally stable silicon oil (to prevent moisture leakage and minimize the compressible gas volume). Pressure sensors were cast at different distances from the heated surface of concrete and the pressure was measured together with the temperature [24,25]. Consistent results were generally obtained in different test conditions (concrete grade, moisture content, heating rate) and values as high as 4–5 MPa were reported in the case of HPC [25], while lower values were reported for normal-strength concrete [3]. For a better understanding of spalling mechanisms, the results of pore pressure measurements should be directly compared with the moisture profiles inside the sample during thermal loading, which might in particular reveal the formation of moisture clogs. First attempts in this direction have been recently made by combining proton-spin relaxation NMR and pressure sensors [26].

In this study, neutron radiography was applied to investigate the water distribution in mortar samples heated from one side up to 550 °C. The samples were mortar slabs with dimensions of  $25 \times 100 \times 100 \text{ mm}^3$  standing on a heating plate, with the  $100 \times 100 \text{ mm}^2$  face exposed to the neutron beam (Fig. 1b). In addition, the temperature distribution within the samples was obtained by means of a series of thermocouples and the vapor pressure at two different locations was measured via pressure sensors [27]. This paper presents this new type of combined measurements and shows some preliminary results. In total, 16 tests were performed on mortar samples at different ages and with different compositions. The results presented in this paper regard two types of high performance mortar. The first sample was a mortar with SAP, which is expected to be prone to explosive spalling due to high moisture content (see [12,28]), while the second sample was a mortar with a combination of SAP and PP-fibers, where the risk of spalling should be reduced [18].

## 2. Materials and test set-up

### 2.1. Mixing and sample preparation

The cross-section of the samples was chosen to fit into the field of view of the neutron beam, which was  $150 \times 150 \text{ mm}^2$ . Thin samples are preferred because they do not induce too large neutron attenuation, thus avoiding the need of long exposure time of the camera to achieve a satisfactory signal-to-noise ratio, and limit artifacts due to sample scattering. At the same time, the samples needed to be thick enough compared to aggregate size, especially to accommodate the pressure sensor heads, which were embedded in the samples during casting. Moreover, thick samples should allow minimizing the boundary effects on the moisture profile along the plate thickness (direction perpendicular to the observed plane). Based on these constraints, mortar samples with dimensions of  $100 \times 100 \times 25 \text{ mm}^3$  were cast.

High-performance mortars with w/b equal to 0.34 were mixed using a 5-l Hobart mixer (the mix composition is presented in Table 1). The binder was Portland cement CEM I 52.5R blended with undensified

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