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Analysis of the effect of heating and re-curing on the microstructure of high-strength concrete using X-ray CT

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HIGHLIGHTS

- We used X-ray CT to examine the effect of heating and re-curing on microstructure.
- Heating caused an increase in total pore space due to an increase in connectivity.
- Cracks in the mortar–aggregate interface and bridging cracks occurred due to heating.
- Water re-curing was effective for reducing connectivity and recovering microstructure.
- Most microstructure recovery occurred within 7 days of water re-curing.

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ABSTRACT

Microstructure recovery plays an important role in restoring the performance of concrete damaged by fire. This research applied X-ray CT and image analysis techniques to non-destructively examine the internal microstructure of high-strength concrete in order to clarify the effects of heating and re-curing on microstructure characteristics. Heating caused an increase in total pore space due to an increase in connectivity caused by the formation of cracks in the mortar–aggregate interface and bridging cracks. Water re-curing, however, was effective in recovering microstructure through the reduction in connected pore space, with most recovery occurring within 7 days.

1. Introduction

Although concrete generally exhibits good fire resistance, exposure to high temperatures can lead to a reduction in overall structural performance including decreased load-carrying capacity, durability and fire resistance. These are caused primarily by chemical changes in the cement paste as well as incompatibility in the thermal behavior of the mortar matrix and coarse aggregates, which lead to weakened matrix strength, coarsened pore structure, and extensive cracking [1]. High-strength concrete in particular has been found to be more susceptible to loss of strength and durability than normal-strength concrete, as well as more likely to suffer from explosive spalling [2,3].

In order to restore safety and performance, repair operations that involve the removal of the damaged concrete and the casting of a patching material are necessary [4]. However, energy and labor costs, as well as waste generation and resource consumption, could be reduced if the performance of the fire-damaged concrete could be restored without the need for labor-intensive operations. Past research works have shown that the re-curing of fire-damaged concrete in water or a high humidity environment can lead to the recovery of strength and durability. This recovery has been attributed to the reduction of pore space and regeneration of hydration products from the rehydration of calcium oxide as well as the hydration of unhydrated cement particles [5–7]. Water supply is of particular importance for recovery, as the rate of rehydration is higher in such cases [8]. Furthermore, while high-strength concrete has been found to perform differently under fire loading, it has also been shown to have better recovery under re-curing due to its dense microstructure [8]. Recovery of durability has been attributed to the filling of pore space and healing of cracks as well as the consumption of calcium oxide during rehydration, which reduces the potential for harmful carbonation, but the instability of healed crack areas may limit the strength recovery [9].

Previous studies have made it clear that changes to microstructure properties such as porosity and pore size distribution play an important role in the degradation and recovery of strength and

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durability. These approaches, however, are generally limited in scale and restricted to either quantitative outputs or two-dimensional images. In addition, there is little research which characterizes microstructure changes differentiating between total pore space and isolated and connected pores. Total pore space may have a strong influence on load-carrying properties, whereas permeability may be more dependent upon the characteristics of connected pore space.

X-ray microtomography (X-ray CT) is powerful tool for non-destructively investigating the three-dimensional (3D) microstructure of a material. Through X-ray CT and image analysis techniques, the internal pore space of a specimen can be qualitatively and quantitatively analyzed in three dimensions and pores characterized as either isolated or part of a connected pore cluster. Furthermore, imaging may be carried out with specimens composed of cement paste, mortar, or even concrete, thus enabling the examination of composite systems. In addition, as the imaging is non-destructive, the effects of various conditions or tests may be carried out on the same specimen with the same material composition. Considering the strengths of this tool, the objective of this research is to utilize X-ray CT and image analysis techniques to clarify the effect of heating and re-curing on high-strength concrete through characterization of the 3D internal microstructure. Specifically, the effectiveness of water re-curing for recovering the microstructure of fire-damaged high-strength concrete was examined by extraction and quantification of the total, isolated, and connected pore space and compared to the case of air re-curing, which represents a control condition.

2. Experimental program

An overview of the experimental program is shown in Fig. 1. Each step of the program will be introduced in detail in the following sections.

2.1. Specimen preparation and curing

Concrete was prepared with a water–cement ratio (W/C) of 0.30, a very low W/C typical of high-strength cementitious materials. The complete concrete mix proportions are given in Table 1. Ordinary Portland cement (C) was used as the binder, along with fine (S) and coarse (G) aggregates from the Shizunai River and super plasticizer (SP) and anti-foaming (AF) admixtures for meeting workability requirements. The workability of the fresh concrete was evaluated using the slump flow and air content, which were measured following the (Japanese Industrial Standard) JIS A 1150 and JIS A 1128, respectively. The target slump flow was 45 cm and the target air content was 5%.

After casting, four cylinders (100 × 200 mm) were sealed and cured in the molds for 24 h, then removed and placed in water curing at 20 °C for four weeks. A single cylinder was then removed from water curing and a 20 mm core was extracted from the center of the cylinder. This core was cut into two 20 mm segments, which were then returned to water curing for another nine weeks. Total curing time from casting to heating was 13 weeks (91 days) in order to achieve a high degree of hydration similar to that of concrete structures in service. The 91-day compressive strength, which was measured using three standard cylinders from the same concrete mix, was 67.5 MPa.

2.2. Heating and re-curing

Fire exposure was simulated using an electric furnace with a temperature control program. The rate of heat increase was set at 10 °C per minute until the target exposure temperature of 600 °C was reached, after which it was maintained for 1 h. The target exposure temperature of 600 °C was selected because the dehydration of calcium hydroxide occurs between 450 °C and 550 °C and produces calcium oxide [10], the rehydration of which has been shown to contribute to the recovery of strength and durability performance under re-curing [8]. Therefore, this temperature enabled the examination of the case in which high degrees of both dehydration and rehydration would occur.

After the conclusion of the high temperature exposure, the door of the furnace was opened and the specimens were allowed to cool in the furnace until the internal furnace temperature dropped below 100 °C. Specimens were then removed from the furnace and cooled at room temperature for approximately 1 h before being placed in one of two re-curing conditions. Air re-cured specimens were placed in a temperature-controlled environment at 20 °C, whereas water re-cured specimens were placed in conditions similar to the initial curing period. Re-curing was carried out for one and four weeks.

2.3. Image acquisition using X-ray microtomography

As summarized by Promentilla and Sugiyama [11] and Landis and Keane [12], the concept of X-ray microtomography is similar to that of Computed Axial Tomography (CAT or CT) scans in the medical field, in which a 3D digital image is reconstructed from a series of two-dimensional (2D) images or "slices." Each voxel (3D pixel) within the 3D digital image has an associated X-ray absorption value that can be correlated to material density, and thus the internal structure can be determined based on the arrangement of the voxels in a 3D space. The resolution of the image can vary from the sub-micron scale or a few microns (for CT systems using synchrotron radiation with a parallel and monochromatic beam) to tens of microns (for microfocus radiation with a cone beam). There is, however, a trade-off, in that the maximum sample size for the higher-resolution systems is limited to less than a few millimeters, whereas specimens on the scale of a few centimeters may be used with lower resolution systems. In the concrete field, X-ray microtomography has been applied at a variety of scales and to a variety of research areas, including pore structure characterization, freeze–thaw damage, sulfate attack, damage evolution, and diffusivity in cracks [11,13–18].

In this research, a desktop microfocus CT system was used for acquiring the slice images. The set-up consists of a microfocus X-ray emitter, a rotation table which allows for 360° imaging, an image intensifier detector with CCD camera, and an image processing unit. Image acquisition was carried out before heating (water re-cured specimen only), after heating, and after one and four weeks of re-curing in water and air. As illustrated in Fig. 2, the focus area for data acquisition was approximately 11.5 mm in height, 20.0 mm in diameter, and roughly centered on the specimen. In this area, 351 slices were obtained. Each slice was 1024 by 1024 pixels in size, with each pixel 22 μm by 22 μm. After image processing and scaling, the voxel size was 22 by 22 by 22 microns.

3. Results

3.1. Cross-sectional images of the concrete specimens

Fig. 3 shows the cross-sectional images of both concrete specimens after heating. In these images, lighter shades of grey indicate higher density materials, whereas darker shades of grey or black indicate lower density materials or air voids, respectively. In the specimen used to investigate water re-curing, it can be seen that there are several large aggregates occupying various spaces; in particular, some of the aggregates are at the surface of the specimen and were likely cut when the core was extracted from the original concrete cylinder. The shades of these aggregates also vary, which implies that the aggregates in the concrete are of differing mineral compositions. As the thermal behavior of aggregates depends on the mineral composition [7], these images clarify that the concrete specimen, when under heating, will undergo varying degrees of thermal stresses.

For the specimen used to investigate air re-curing, one large aggregate appears to occupy the center of the specimen at lower heights, whereas other smaller aggregates can be seen in the upper

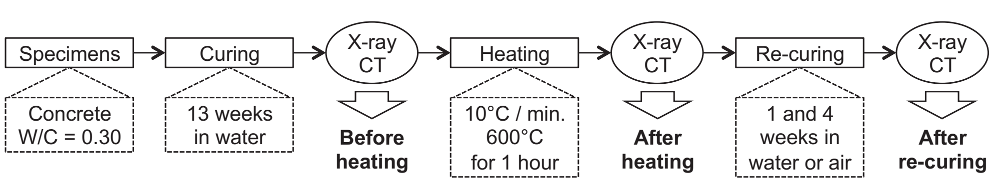

Fig. 1. Overview of the experimental program.