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Freeze-thaw crack determination in cementitious materials using 3D X-ray computed tomography and acoustic emission☆

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ABSTRACT

As concrete freezes and thaws cracks may develop. These cracks can provide a path for water and ionic species to penetrate the concrete. This may reduce the service-life of the concrete element. In this study, X-ray computed tomography (CT) was used as a non-destructive technique to characterize the microstructure of mortar samples that were exposed to different levels of freeze-thaw damage by varying degree of saturation in the samples (75, 90, 95, and 100% degrees of saturation). Acoustic emission (AE) experiments were performed during freezing and thawing to investigate sample cracking behavior. The volume of cracks present within the mortar samples after freezing and thawing were determined using X-ray CT and compared to passive acoustic emission data. The location/source of cracks was also determined using X-ray CT. The crack sources (i.e., void, aggregate, interfacial transition zone, or paste) were determined using X-ray CT and were related to AE activities during cracking. Crack volumes were found to increase with increased levels of saturation, and visual observations of cracking were found to correlate with AE signatures of various crack sources.

1. Introduction

The cracking behavior of a composite system like concrete is dependent on the properties of its constituent components. Cracks can form within a concrete specimen in a variety of ways, such as an applied external force, temperature changes leading to volumetric mismatch, and internal chemical reactions usually caused by the ingress of aggressive chemicals [1,2]. Small cracks can form and eventually propagate, thus serving as a source for the development of larger cracks [2,3]. A number of nondestructive techniques have been used to better analyze cracking within concrete, such as visual inspection, microscopic analysis, 3-D X-ray computed tomography (CT), ultrasonic pulse velocity, resonant frequency, and acoustic emission [4,5]. Acoustic emission (AE) is a real-time, non-destructive evaluation method that can be used to investigate cracking in concrete [6,7]. Passive AE corresponds to soundwaves that are produced when cracks develop in a material. When a crack is formed in the concrete, energy is released and a portion of this energy is dissipated as an acoustic wave with specific waveform signatures [8–10]. Acoustic waves and corresponding energy could be released by the formation of cracks or microcracks due to freeze-thaw cycles [11–13]. The characteristics of the acoustic waves (e.g., the number of counts, number of hits, amplitude, rise time, duration, velocity, frequency, signal strength, and corresponding AE energy) may provide a method to evaluate cracking in samples exposed to freezing and thawing [3,14]. Generally, the strength of the signals and the waveform properties depend on the amount of released energy, distance, source, and the orientation of the source with respect to the AE sensor [15]. Passive AE tests involve AE transducers that continuously sense acoustic waves generated by the formation of cracks or microcracks during a test. These acoustic waves are then analyzed to identify the type and magnitude of the resulting damage. In cementitious materials, the source of AE activities may be located within different constituents consisting of the aggregate or the matrix (cement paste and interfacial transition zones (ITZs)), and cracking in each constituent may create different types of AE activity that can be used to classify cracking type and phase location [15].

3D X-ray CT can also be leveraged as an efficient non-destructive characterization of the microstructure of mortar

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samples [16]. This method allows the user to visualize the components present within an opaque subject being analyzed as well as to obtain accurate quantitative information on the area and volume of specific components [16,17]. Additionally, X-ray micro-CT scans can be assembled into 3D models and used for finite element computations of fracture in mortar and concrete [18–21] The images taken consist of various grey intensities that correspond to different densities within the sample, where darker shades usually correspond to low density components (i.e., voids, cracks) and lighter shades usually correspond to higher density materials (i.e., cement paste, aggregate) [16,22]. To identify air voids, a threshold intensity value can be chosen. Using a threshold value that corresponds to a gray intensity, the original image can be transformed to a binary image, which can then be used to measure the size of the components that is represented by the remaining pixels in the binary image [16,23].

The purpose of this work is to characterize the fracture behavior of the concrete by investigating the AE waveforms and comparing AE activities to visual observations obtained from 3-D X-ray computed tomography (CT). Accordingly, the fracture behavior of the individual components of the concrete (e.g., within the cement paste, aggregate and ITZ) can be identified and characterized due to freeze-thaw damage.

2. Experimental program

2.1. Materials and mixture proportioning

All tests were performed on a mortar mixture with a water-to-cement ratio (w/c) of 0.42 by mass and a sand volume fraction of 55%. The mortar contained ordinary Type I Portland cement (OPC) with a fineness of 375 m^2/kg ($C_3S = 60\%$, $C_3S = 10\%$, $C_3A = 9\%$, $C_4AF = 10\%$, and $Na_2O_{EQ} = 0.86\%$). The aggregate was natural sand with a maximum size of 4.75 mm, specific gravity of 2.61, fineness modulus of 2.89, and an absorption value of 2.2% by mass. ASTM C305-12 [24] was followed during the preparation of the samples in a standard mortar mixer. The mortar samples with dimensions of 25.4 mm × 25.4 mm x 300 mm (1 in x 1 in x 11.81 in) were cast, vibrated, and sealed for 28 d. Following curing, mortar samples were cored and cut into cylinders with a 10-mm diameter and a 20-mm length for freeze-thaw test.

The samples were placed in a vacuum oven at 50 °C ± 1 °C with a pressure of 2.66 kPa ± 0.66 kPa (20 mmHg ± 5 mmHg) for one day. The samples were placed in a desiccator using two small spacers underneath each sample to provide a small gap between the lower surface of the sample and the bottom of the container. The samples were evacuated to a pressure of 10 ± 5 mmHg for 3 h. Following evacuation but still under vacuum, de-aerated water (de-aerated by vacuuming the water for 15 min) was added to the container until the sample was completely submerged. The sample remained submerged for 1 h. Next, the submerged sample in the water was transferred to a 23 °C ± 0.5 °C chamber and remained in the chamber before testing for a period of 3 d. This condition was defined as having a 100% degree of saturation (DOS). The samples to be used for testing at lower degrees of saturation were submerged for 3 d and were then removed from the solution. The samples were set aside to dry while monitoring their mass every 1 min–5 min until the desired mass (i.e., degree of saturation) was obtained to reach the target DOS. Samples were placed in a double sealed bag for a week prior to freeze–thaw testing to assure uniform distribution of moisture within the sample. Samples with 75%, 90%, 95%, and 100% DOS were prepared in this study. The DOS was calculated by using the ratio of the volume of absorbed water to the total volume of water that the sample was capable of absorbing.

2.2. Freeze-thaw testing procedure

Freeze–thaw testing was performed on samples using a thermally insulated setup shown in Fig. 1. The temperature of the sample was controlled using a cold plate. One AE sensor was used to investigate the acoustic activity of a sample during freezing and thawing. Vacuum grease was used to attach the sensor to the sample. High vacuum silicone grease (polydimethylsiloxane, amorphous silica, dimethyl siloxane, and hydroxyl) was used that good resistance to water, chemicals, and high and low temperatures, and it was found to be stable over the temperature range of the test. A single cooling and heating cycle was applied using a cold plate whose temperature was controlled as described in Fig. 1c, with a cooling rate of 2 °C/h and a heating rate of 4 °C/h (Fig. 1c). With this setup, one-dimensional heat flow was produced using a heat sink (i.e., cold plate) and an insulated enclosure. Thermocouples were used to monitor temperature in three locations (shown in Fig. 1): on the surface of the cold plate, at the bottom of the mortar sample, and around the middle of the sample. Temperature sampling was performed at a rate of 0.05 kHz during the thermal cycle.

The acoustic emission (AE) activity produced by generation of cracks or microcracks was monitored during freeze-thaw testing. The acoustic events refer to sound waves that are produced when a material undergoes cracking, resulting in stress waves because of the energy release in a material. The detected AE can cover a wide range of inaudible and audible frequencies [25]. Therefore, a threshold value of 45 dB_{AE} was selected to filter any noise created during test. A VS375-M[1] cylindrical piezoelectric sensor (with a frequency range between 100 kHz and 700 kHz) was used to convert the captured acoustic waves into electrical signals [26] The VS375-M was found to be a promising sensor in capturing internal cracking in cementitious materials from previous works [12,15]. The AE filtered signals through AE sensor were amplified and recorded in a data-acquisition system. A Vallen AMSY-5 acoustic emission system with the capability of wave transient recording (TR) was used [27]. Therefore, a complete waveform diagram of any captured wave could be recorded and then analyzed. The area under the absolute value of the volt-time response of the AE sensor during an AE event was calculated and considered as the "AE signal energy" that is released. This AE signal energy is proportional to the fracture energy [12,28,29]. Verification of the AE signal processor, parametric input channels and system performance are reported in Ref. [30] according to EN 13477–2:2010 [31].

2.3. 3D X-Ray micro-computed tomography (CT) procedure

Crack and void distributions within mortar samples due to freeze-thaw cycling were monitored using an X-ray micro-CT instrument. X-ray micro-CT is useful in visualizing features in the interior of solid objects so that their 3D geometry and properties can be obtained and measured accurately. The components of this measurement consist of a source and a detector, with the test specimen placed on a rotating stage between the two [16]. Before scanning, samples were first dried in a vacuum oven at 50 °C ± 1 °C and a pressure of 20 mmHg ± 5 mmHg for a day after freeze-thaw testing in order to remove the moisture for easier X-ray imaging and preventing further hydration until X-ray

[1] Certain commercial equipment and/or materials are identified in this report in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment and/or materials used are necessarily the best available for the purpose.