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Experimental and numerical study of crack behaviour for capsule-based self-healing cementitious materials

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HIGHLIGHTS

- Crack behavior of capsule-based self-healing cementitious material was studied.
- A 2D lattice model was used to simulate the crack process.
- A real phase-segmented 3D structure was constructed based on 2D XCT images.
- Fracture behavior was compared between real structure and 3D lattice model.

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ABSTRACT

A 2D lattice model was constructed to simulate crack process of in shell-interlayer-cement paste zone. The simulated tensile strength was validated by an experimental uniaxial tensile test and used as the input for a 3D lattice model, which was constructed to perform mechanical analysis based on a series of 2D X-ray microtomography images. The fracture behaviour of the 3D lattice model with assigned mechanical properties gave a similar crack pattern and tensile strength as the real structure. This study is expected to provide a feasible approach for investigating the fracture and trigger behaviour miccrocapsules embedded in a self-healing cementitious system.

1. Introduction

Microcracks often develop in cementitious composites as a result of mechanical loading, environmental loading (e.g., freezing and thawing, rebar corrosion), and volumetric instability (e.g., shrinkage in fresh or hardened concrete, thermal contraction). Once formed, they are extremely difficult to be detected and repaired by conventional methods before they develop, coalesce and grow into macrocracks. Therefore, cracking related deterioration of cementitious materials has been recognized as a severe threat to the safety, integrity and durability of concrete structures. In recent years, inspired from biological self-curing phenomenon, the microcapsule based self-healing concept has attracted substantial attention and showed its great potential for the application in cementitious materials [1–7]. In such applications, healing agents are first encapsulated in microcapsules, which are then incorporated in a fresh cement-based mixture. Once the incorporated microcapsules are ruptured by the propagation of cracks or other stimuli, the self-healing is realised through the release and reaction of repairing chemicals in the region of damage.

As an essential step in the self-healing process, the crack-induced rupture behaviour of microcapsules plays a decisive role in realizing the self-healing function. It is generally believed that the bonding strength, the size parameter and the mechanical properties (e.g., elastic modulus, strength) of microcapsule are among the important factors greatly influencing the self-healing performance as well as the mechanical properties of cementitious composites [8,9]. Thus, a proper selection and design of the microcapsules could offer a higher healing efficiency with lower degradation of mechanical properties of the cementitious systems.

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In general, there are two available routes to design/prepare the microcapsules with desired properties. The first route is using an experimental method. Some experiments methods including morphology observation, crack surface inspection and mechanical recover rate calculation, have been performed on microcapsule-embedded cementitious materials [10–13]. Microcapsules with desired chemical and physical properties could be obtained in such a way on the basis of the experimental results. However, with this experimental method the analysis is only done after the test which sometimes can only provide limited information from the existing test. It cannot give an indication on what properties the microcapsules should have in order to be prone to rupture by concrete cracking and to further predict how the embedded microcapsules will influence the self-healing behaviour in a broader sense. The second route is predicting the relevant parameters of the microcapsules through simulation approaches. There exists a wide range of micromechanical models for predicting the optimal dosage and parameters of the microcapsules as well as the mechanical properties of the cementitious composites. Most of these models use a continuum approach to predict the elastic properties of this three-component composite [14,15], it is inherently incapable when dealing with fracture process in this type of materials. Moreover, the bonding strength between shell materials and cement paste is often ignored resulting in an overestimation of the simulated values.

In order to simulate fracture behaviour of microcapsule-embedded cementitious materials in a more realistic way, two points should be considered: 1) The first point is related to obtaining the mechanical parameters of the components of self-healing composite, corresponding to the shell, cementitious matrix and interlayer, respectively. These data can be used directly as inputs for the 3D modelling of the self-healing system. Similar to the fact that the interfacial transition zone (ITZ) in concrete has a great influence on the mechanical properties of concrete [16], the interlayer properties between shell materials of microcapsules and the cementitious matrix also significantly affect the stability of the microcapsules, the potential self-healing efficiency and the mechanical properties of cementitious structure. However, studies regarding micromechanical properties of the interlayer between microcapsules and matrix such as bonding strength and elastic modulus are very limited. In this context, fundamental and practical research is essentially needed for the application of capsule-based self-healing cementitious materials. 2) The second point is obtaining a real 3D microstructure of microcapsule-embedded cementitious materials, in which the shell thickness, particle size and distribution of microcapsules should be correlated with the real situation. Micro X-ray computed tomography (XCT) offers a non-destructive experimental technique, which has already been used to collect the microstructure information of cement paste in terms of digitized voxels [17]. By segmenting the phases and defining the local mechanical properties to individual phases, a 3D lattice model can be formed [18].

In this study, the nanoindentation method was used for mapping the mechanical properties of complex surfaces of shell-interlayer-matrix. The measured properties were directly applied as inputs for constructing a 2D numerical model. A direct tension test was simulated and the bonding strength and the elastic modulus of the interlayer between shell and matrix were obtained based on the 2D model. The XCT technology was further used to get the 3D volume images of an actual cementitious material in which capsules were incorporated. Based on these images and the mechanical information obtained from the 2D model, a 3D lattice model of a capsule-based self-healing cementitious material was formed. The crack pattern and the self-healing behaviour of the capsules-based self-healing cementitious materials were discussed accordingly.

2. Materials and methods

2.1. Materials and sample preparation

2.1.1. Materials

CEMⅠ42.5N Portland cement in accordance with European standard EN-197-1 and deionized water were used. The phenol-formaldehyde (PF) microcapsules (mean diameter 201 μm) were prepared according to our previous study [19].

2.1.2. Sample preparation for 2D SIC zone study

The shell material i.e., PF resin as shown in Fig. 1a, was synthesised under the same condition as that of the microcapsules. After that, the PF resin bulk was cut into small pieces of 10 mm × 10 mm × 3 mm. Cement paste with water-to-cement ratio of 0.4 was prepared and cast into the 10 mm × 10 mm × 10 mm prismatic moulds. Then a piece of shell material (10 mm × 10 mm × 3 mm) was placed on the top of cement paste sample. A vibration plate was used to minimize the amount of entrapped air in cement paste and to maximize the contact area between two materials. After curing at room temperature in the lab for 24 h, the specimens were move to a curing room at 23 °C and over 95% relative humidity. After curing for 28 days, the samples were taken out from the fog room and demoulded. Four samples were prepared in total, in which three samples were prepared for tensile test (Section 2.2.2) and the rest one was sealed by epoxy resin for nanoindentation to obtain the local mechanical properties of shell-interlayer-cement paste (SIC) zone. One of the side surfaces of the epoxy sealed sample containing both shell material and cement paste was then ground with No. 2000 and 4000 emery paper and subsequently polished with 6 μm, 1 μm and 0.25 μm diameter paste on a lapping table. The samples were further cleaned in an ultrasonic bath to remove any residues. Fig. 1b shows the prepared specimen for nanoindentation test.

2.1.3. Sample preparation of microcapsule-based system for 3D crack behaviour study

The microcapsules-embedded cement paste specimens for XCT study were prepared with a water-to-cement ratio of 0.4 and contained 4% PF/DCPD microcapsules (mean diameter 201 μm) of cement mass. The microcapsules were blended with cement and deionized water and mixed for one minute to achieve a good workability. After mixing, the fresh mixture was cast into a 6.7 mm × 13.4 mm plastic cylindrical mould. Then they were carefully compacted on a vibrating table to reduce the entrapped air. The specimens were demoulded after curing under room temperature (RT) and local lab environment for 48 h. Then they were cured in a wet chamber at 23 °C and over 95% relative humidity. After curing for 28 days, samples were taken out from the curing room. Morphology of microcapsules which applied in this study. Fig. 2 shows the optical microscopy images of microcapsules we used in this study and an example of the triggered microcapsules embedded in cement paste. Four samples were prepared in total, in which three samples for tensile test and one sample for XCT scanning.

2.2. Test method

2.2.1. Nanoindentation

Nanoindentation tests were performed to obtain the local micromechanical properties of SIC complex using Agilent Nano Indenter (G200, Keysight, USA) with a diamond Berkovich tip. The continuous stiffness method (CSM) developed by Oliver and Pharr [20] was used in this study. By this method, a small oscillation is imposed on the primary loading signal and the response of