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Towards understanding stochastic fracture performance of cement paste at micro length scale based on numerical simulation

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HIGHLIGHTS

- The stochastic micromechanical properties of cement paste were predicted.
- Results show good agreement with data in the literature.
- Specimen with lower w/c ratio has higher and less variable strength and elasticity.
- A strong size effect exists in the modulus/stress ratio of cement paste.

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ABSTRACT

This work presents a study of stochastic fracture properties of cement paste at the micro length scale based on a combination of X-ray computed tomography (XCT) technique and discrete lattice type fracture model. Thirty virtual specimens consisting of pore, outer hydration products, inner hydration products and anhydrous cement particles were extracted from 3D images obtained through XCT from real cement paste samples. These virtual specimens were subjected to a computational uniaxial tension test to calculate their tensile strengths and elastic moduli. The predicted stochastic strengths were analysed using Weibull statistics, showing that specimens with lower w/c ratio yield higher strength and less variability. The strength-porosity and modulus-porosity relations were investigated based on existing empirical models. It was shown that existing models can predict the properties in the studied porosity range quite accurately, with the exponential model having the highest determination coefficient among all the models for both relations. Finally, by comparing the existing data in the literate, it is found that the smaller cement paste specimens have higher modulus/tensile strength ratio, which indicates that they are able to have more strain at the peak load.

1. Introduction

Cement paste is a porous and heterogeneous material [1]. As a basic binding material in concrete, it has generated considerable research interest. It is generally accepted that fracture of conventional concrete starts from micro-cracks in the cement paste where local tensile stress exceeds its tensile strength. Understanding the deformation and fracture performance (i.e. tensile strength and elastic modulus) of cement paste at the micro-scale is therefore of significant practical importance and scientific interest.

Nanoindentation has been utilized for quantification of local properties such as elasticity and hardness of micro level components in the matrix [2–6] for a long time. Based on the same principles, peak-force tapping atomic force microscopy (AFM) can be applied as an alternative tool to quantify the local elastic properties [7]. These techniques provide a meaningful experimental input for analytical and numerical models used to calculate the global micromechanical properties of cement paste [8–13] which can be further used as input within a multi-scale framework to simulate the macroscopic mechanical performance of concrete [14–16].

Although a lot of valuable micromechanical information was obtained to set a basis for understanding and improving the macroscopic mechanical performances, stochastic micromechanical properties of cement paste have been rarely studied due to the complex and time-consuming modelling procedure. Furthermore, for a number of reasons that include problems with producing and measuring miniaturized mechanical samples, the predicted mechanical properties are difficult to verify experimentally at the micro scale. As reported in [17,18], a pioneering work on experimental micromechanics of cement paste has been conducted at the Delft University of Technology. Micrometre scale specimens

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with a cubic dimension of 100 μm were produced by a micro dicing saw and ruptured by a diamond wedge tip mounted on a nanoindentation system. The splitting tensile strength of the tested specimens was derived from the recorded critical load and the test results show a large dispersion, as expected for a highly heterogeneous material at this scale. For a specific ruptured specimen, detailed microstructure information cannot be obtained due to technical and instrumental limitations. Therefore, it is hard to correlate the fracture properties with its microstructure for quantitative assessment at the micro scale. Instead, application of a microstructure-informed numerical model [12,13,19,20] offers an opportunity to achieve this. Such a model requires a detailed microstructure and micromechanical properties of individual components in the material. The 3D microstructure can be obtained either by modelling or experiments. Although numerical cement hydration models have clear advantages in terms of time effort and ease of obtaining, cement particles are commonly modelled as spherical in such models [21,22]. This has an influence on the simulated hydration of cement [23]. Furthermore, it is reported that the assumed morphology of hydrates in the simulated microstructure significantly influences micromechanics-based elastic stiffness estimates of cement paste, particularly at very early age [24]. Furthermore, it is worth mentioning that a popular digital microstructural model CemHyd3D [25] permits a direct representation of multiphase, multi-size and non-spherical cement particles using SEM images. However, it is reported by Hain and Wriggers [26] that, because of random based rules, the parts of microstructure in CemHyd3D are distributed very evenly. In particular, there are no accumulations of pores in its simulated microstructure as observed in X-ray computed tomography (XCT), which is now becoming a widely used technique for three-dimensional microstructure characterisation of cement-based materials [27–29]. The basic idea of XCT is simple [30]: rotate a material under X-ray scanning and collect the absorbed X-ray on a detector to create a series 2D projections from which the density spatial distribution of phases inside the material can be visualised by greyscale levels. In terms of cement paste, pores have the lowest greyscale level, while anhydrous cement grains have the highest greyscale level. Based on the histogram of greyscale levels, image segmentation can be conducted to isolate different components, namely pore, inner and outer hydration products, and anhydrous cement grains. Note that, as reported in [31], C–S–H and nano-CH are associated, not merely as a simple biphase mixture. For simplification, the inner and outer hydration products in the current study are referred to inner (high density) and outer (low density) C-S-H gel, respectively. In order to show the stochastic mechanical properties of cement paste at micro-scale, for each w/c ratio (0.3, 0.4 and 0.5), ten microstructures with a cubic dimension of 100 μm^3 were sliced from XCT images of cement paste specimens cured for 28-days. These microstructures were then used as input for fracture simulations performed using a lattice type fracture model. The computational uniaxial tension test was performed on three different loading directions for each cubic specimen, making it 30 simulations for each w/c.

Lattice type fracture model is one of the most popular microstructure informed numerical models to study fracture process and stress-strain response of cement-based materials. Its main strength is the simulated detailed crack patterns which resemble experimental observations very closely [32–35]. As an experimental procedure at the micro scale has been designed and utilized to calibrate the input parameters in this model recently, the predicted micromechanical properties of cement paste are expected to be more reliable [13] compared to previous research. Therefore, lattice fracture model is adopted in this work to simulate the fracture behaviour of cement paste based on XCT obtained microstructure. For better understanding of its stochastic fracture performance, a Weibull statistical analysis is conducted to analyse the simulation results. In addition, several widely used empirical equations describing the effects of porosity on mechanical properties for cement-based materials were examined and extended to the cement paste at micro-scale on the basis of the simulated results.

2. Virtual specimen generation

2.1. X-ray CT scanning

In the experimental program, small cement paste prisms with cubic cross-section of 400 μm × 400 μm and length of 1 mm were produced and scanned by a microcomputed tomography system. Cement pastes were prepared with standard grade CEMI 42.5 N Portland cement and deionized water. The w/c ratios of used paste were 0.3, 0.4 and 0.5.

The cement and deionized water were carefully mixed with the designed w/c ratio, vibrated and poured into a PVC cylinder (diameter, 24 mm, height 39 mm). To prevent bleeding, the cylinder was rotated with a speed of 2.5 revolutions per minute for 24 h. The specimens were sealed in the cylinder and stored in a room with temperature of 20 °C. After sealed curing for 28 days, the specimens were demoulded and a slice with a thickness of 2 mm in the middle part was cut out using a precision diamond saw. The slice was then attached on the glass substrate using a UV bonding resin and grinded down to 400 μm by diamond ring grinding discs with grit size of 125 μm and 30 μm in descending order. To ensure that both surfaces of the slice are smooth, the slice was detached when reaching 1 mm thickness, flipped over and reattached on the glass substrate. The same grinding procedure was performed on the new surface. A micro dicing saw was then used to cut through the slice to produce small prisms with a square cross section of 400 × 400 μm^2 as presented in Fig. 1a. The back-scattered electron (BSE) image of the small prism is shown in Fig. 1b.

The small prism was clamped by a special holder (Fig. 2) and fixed in the rotatable stage of a high resolution XCT scanner (Phoenix Nanotom, Boston, MA, USA) for acquiring raw greyscale images with a source voltage of 120 kv and current of 60 μA. The sample was rotated 360° and, in total, 2880 shadow projections with a pixel size of 0.5 μm were acquired on the digital GE DXR detector (3072 × 2400 pixels). Each projection image was averaged with an exposure time of 6 s. The reconstruction work was conducted by the Phoenix Datos|x software. To reduce the influence of beam hardening in the XCT experiment, a cuboid region of interest (ROI) with a cross-section of 200 μm × 200 μm and length of 500 μm was extracted from the middle region of the specimen for the analysis (Fig. 2b). The mesh discretization in the fracture model is correlated with the voxel size from the material structure, and in total 90 computational uniaxial tensile tests were to be performed, which means a huge computational effort were needed for this study. For this reason, the resolution was reduced to 2 μm^3/voxel through a "median method" implemented in the X-ray reconstruction software. Finally, a 3D stack of 8-bit cross-section images were generated.

2.2. Microstructure characterisation

Quite a few image segmentation methods have been suggested in the literature. Herein, a global thresholding method is applied [29,36,37]. As shown in Fig. 3, based on the greyscale-level histogram, four threshold greyscale levels were defined to segment pores, anhydrous cement grains, inner hydration products and outer hydration products from the raw grey images. The reader is referred to [13] for further information on the image segmentation, only a short description is addressed herein: (1) T_1, the