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Experimentally validated multi-scale modelling scheme of deformation and fracture of cement paste

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ABSTRACT

This paper presents a validation procedure of multi-scale modelling scheme by making, testing and modelling deformation and fracture of cement paste beam at sub-*meso* scale (between micro and meso scale). Miniaturized three-point bending testing concepts were adopted. Cement paste beams with a cross-section of 500 μm × 500 μm were fabricated using a micro dicing saw and subjected to bending using nanoindenter. Simultaneously, fracture behaviour of the same size specimens was predicted by a microstructure informed multi-scale lattice fracture model in which input microstructures are obtained by X-ray computed tomography at two scales (micro and sub-*meso*). Uncoupled upscaling approach was used to relate the material's mechanical behaviour at sub-meso scale with the microstructures at lower scale. A good agreement between experimental and numerical results was observed which shows that starting from micro scale and with relatively simple mechanical considerations it is possible to correctly reproduce behaviour at upper scale.

1. Introduction

As the most widely used construction material in the world, concrete has generated considerable research interest. One of the most concerned issue involves mechanical properties characterisation, i.e. strength and modulus, which are the basic information for concrete structure design and durability investigation. Since it is well recognised that concrete is a fairly heterogeneous material ranging from nanometres to millimetres [1], in pursuit of better understanding of local mechanical properties and the mechanisms determining the materials' behaviour, multi-scale approach [2–5] has been devoted to the investigation on mechanical properties on different length scales. Commonly, three scales can be considered, namely micro, *meso* and macro scale, roughly corresponding to the feature microstructures of cement paste, mortar, concrete respectively. At the micro scale (few μm to hundreds μm), cement paste is a heterogeneous and complex porous medium. At the meso scale, in the range between mm to cm, small sand particles with interfacial transition zone (ITZ) embedded in a cement matrix can be typically observed. At the macro scale, concrete consists of small sand particles bonded by matrix of mortar. The uncoupled upscaling method is commonly applied to bridge any two scales [2,3,6–8]. In the uncoupled upscaling method, the simulated global mechanical performance of composites at lower scale are directly assigned as the local mechanical properties of the matrix in the upper scale models. This method is often criticized because of its simple mechanical considerations. Furthermore, it is not easy to be verified by experiments at upper scale because not only the matrix is regarded as a homogenous material but also the ITZ is introduced in the system, which is indeed another complex issue [9,10]. Therefore, the main aim of this work is to verify the upscaling method through a relatively simple scheme. The verification scale is chosen in between micro scale and *meso* scale in which the cement paste matrix and air voids or big capillary pores are recognised. As the investigated length scale is much smaller than the typical laboratory sample size, the experimental challenges include producing and mechanical testing of such miniaturized samples.

As a high precision instrument that can record small load and displacement with high accuracy and precision, nanoindenter is becoming a general tool in small scale mechanical testing. Utilizing this technique for micro-mechanics provides a basis for the development of various miniaturized testing concepts, e.g. micro-beam bending test [11,12], pillar compression test [13,14] and pillar indentation splitting test [15]. Based on the recorded load and displacement data during the test, the basic deformation mechanisms and behaviour of small mechanical samples can be used to derive elastic, plastic and fracture material properties. Recent applications of micro dicing saw on preparation of small cement paste specimens make it possible to prepare the small scale specimens such as pillar, beam and cylinder [16–18]. In this work,

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miniaturized three-point bending testing concepts were adopted. Cement paste beams with a cross-section of 500 μm × 500 μm were fabricated using the micro dicing saw and subjected to bending using nanoindenter.

In parallel with the experiments, a multi-scale, finite element lattice-based model is introduced here to simulate the deformation and fracture performance of these sub-*meso* scale specimens under three-point bending. Lattice model is one of the most popular approaches used to explain fracture in cement based-materials, mainly because the simulated cracks are very realistic and resemble to a great detail cracks observed in laboratory tests and in practice [19,20]. To consider the heterogeneous and multi-phase microstructure in the simulation, it is essential to have a well-characterized, three-dimensional information about the material. Therefore, three-dimensional X-ray computed tomography (XCT) has been adopted in the current study to characterize the microstructures of this material at two scales. The uncoupled upscaling method [2,3,6–8] is then used in this paper to relate the material's mechanical behaviour at sub-*meso* scale with the microstructures at lower scale. After validation, influence of porosity on the mechanical performance is studied based on the predicted results.

2. Experimental

2.1. Materials

Cement pastes were prepared with standard grade CEM I 42.5 N Portland cement and deionized water. The w/c ratios of used pastes were 0.3, 0.4 and 0.5. After careful mixing, the fresh paste was cast in a plastic cylinder mould of 24 mm diameter and 39 mm height and was carefully compacted on a vibrating table to minimize the amount of entrapped air. The samples were rotated at a speed of 2.5 rpm for 24 h to minimize bleeding, and subsequently cured in sealed conditions at room temperature (20 °C) until 28 days. At the end of the curing period, samples were demoulded and cut into 2 mm slices by a precision saw. The hydration was arrested by solvent exchange method using isopropanol [21]. In order to enable faster water-solvent exchange, slices were immerged five times and taken out for a period of 1 min. Afterwards, each slice was placed for 72 h in isopropanol and subsequently taken out, and solvent was removed by evaporation at ambient conditions.

2.2. Miniaturized beam preparation

The following two steps are needed to produce the micro-beam with a cross-section of 500 μm × 500 μm. The first step is to grind the thickness of cement slice down to 500 μm with flat surfaces. For this purpose, a "Struers SystemAbele Accessories" system was used for both grinding and polishing work. The slice was mounted on a glass slice using a UV bonding resin and then ground down to 1 mm using diamond ring grinding discs with grit size of 125 μm and 30 μm in order. Afterwards, the slice was turned around to continue the grinding procedure until the required thickness (i.e. 500 μm) was reached.

The next step is to cut micro-beams from the prepared thin slice which is achieved by running a micro dicing saw (MicroAce Series 3 Dicing Saw) over the thin section as schematically shown in Fig. 1a. To make sure that the cement paste beams have the required dimensions and that also the glue between cement paste and glass is sliced, the diamond blade is cutting a few micrometres into the glass plate. The micro-beams were deboned from the glass using acetone and observed in environmental scanning electron microscope (ESEM) to check the geometry as shown in Fig. 1b. The final beams all have a cross-section of 500 × 500 ± 5 μm^2.

2.3. Micro-beam three-point bending

Fig. 2a schematically shows the setup of miniaturized three-point bending test. The test was instrumented by Agilent G200 Nano indenter system equipped with a diamond cylindrical wedge indenter tip (radius 9.6 μm, length 700 μm, see Fig. 2b). The micro-beams were placed in flutes of a 3D printed support with a span length of 12 mm (Fig. 2c). A line load was then applied at the centre of the span by the indenter. To observe the fracture process during loading, an external camera was added. A video recording the fracture process is available as Supplementary material.

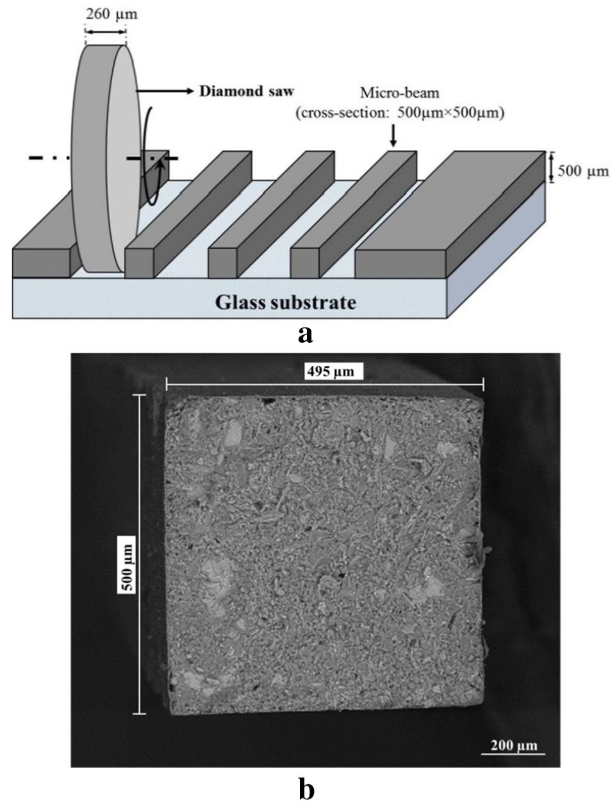

Fig. 1. (a) Schematic view of sample preparation. (b) ESEM image of cross-section of micro-beam.

The experiments were run using displacement control with a loading rate of 500 nm/s. Fig. 3 shows the typical recorded load-displacement diagram in which the maximum force F_{max} was used for the flexural strength calculation using elastic beam theory [22]:

$$f_t = \frac{3F_{max}L}{2d^3} \tag{1}$$

where L is the length of span and d is the cross-sectional dimension of the square. In the evaluation of Young's modulus of the cement paste beams, data of recorded load displacements in the range between 50% and 80% of maximum load were used. The plots in this range are linear to the point of fracture, reflecting the well-known fact that cement paste is a quasi-brittle material [1,23]. Young's modulus E is evaluated as:

$$E = \frac{L^3}{4d^4}\cdot S \tag{2}$$

where S is the average slope in the range between 50% and 80% of maximum load. Since the deflection of the beam cannot be measured directly in the current setup, the recorded vertical displacement of the indenter is used as an estimate, which may lead to a somewhat lower value of modulus since the local imprinting of the indenter into the beams is recorded as well.

2.4. Fracture pattern visualisation

After testing, one failure micro-beam with w/c ratio 0.4 on the