Micromechanical modelling of cement paste using X-ray computed tomography and statistical nanoindentation

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MICROMECHANICAL MODELLING OF CEMENT PASTE USING X-RAY COMPUTED TOMOGRAPHY AND STATISTICAL NANOINDENTATION

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Abstract

This work proposes a new method for micromechanical simulation of cement paste based on a combination of statistical nanoindentation and XCT technique without the need for explicit identification of distinct phases. A linear relationship between the greyscale level and local Young's modulus was assumed and verified by the two-sample Kolmogorov-Smirnov statistic. Based on this assumption, the fracture behaviour of a digital cubic volume (100 µm) under uniaxial tension was simulated using a lattice fracture model. The proposed method was compared with the results obtained from a approach used previously by the authors in which discrete phases were considered. The two methods show similar crack patterns and stress-strain responses. The proposed method is regarded more promising as it captures also the gradient of material properties within the discrete phases.

1. INTRODUCTION

Cement paste is a multi-phase material comprising several phases, most importantly calcium silicate hydrate (C-S-H), calcium hydroxide (CH), anhydrous cement clinker and pores. Consequently, micromechanical models consider, in general, a multi-phase microstructure. As XCT and nanoindention are becoming the general tools in charactering material structure and local micromechanical properties respectively, more and more micromechanical models are carried out based on the information from these two measurements [1-3]. The output from XCT are the greyscale-level based images, while the results from the statistical nanoindentation test are a large number of micromechanical properties of probed locations. To have proper input format for the micromechanical models, a phase segmentation method is generally conducted on the XCT greyscale-level images for a multi-phase microstructure, while the micromechanical properties of induvial phases are extracted by statistical approaches such as the deconvolution method [4-6]. However, it should be noted that phase segmentation is not a standardized technique: many methods exist, and it is difficult, if not impossible, to ascertain which segmentation method produces more accurate results. Furthermore, the scatter in the results of statistical nanoindentation test is big and it is debated in literature whether this method can be used at all for heterogeneous materials like cement paste [4-7].

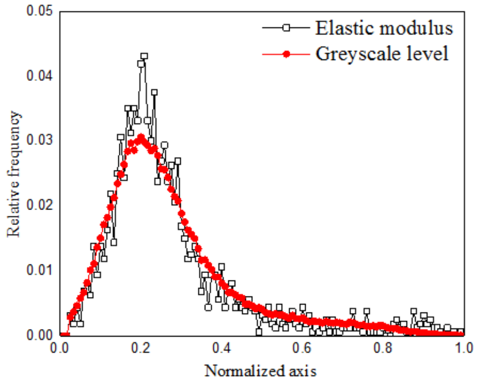
To this end, this work proposes a new method for micromechanical simulations of cement paste based on a combination of statistical nanoindentation and XCT technique without the need for explicit identification of distinct phases. The material structure of cement paste was characterized by XCT. The probability density function (PDF) of micromechanical properties (i.e. histogram of micromechanical properties) was quantified using statistical nanoindentation. Without image segmentation or histogram deconvolution, micromechanical properties were directly correlated with the greyscale level by a linear equation.

2. EXPERIMENTAL

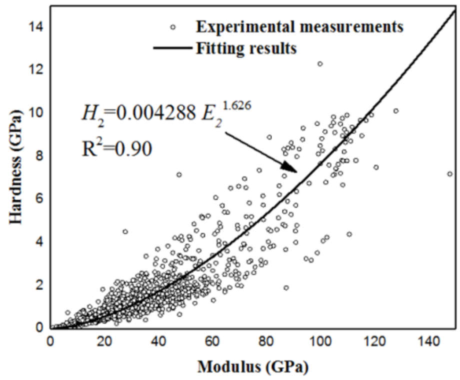
The material used in this work was a standard grade OPC CEM I 42.5 N paste with 0.4 water-to-cement (w/c) ratio, curing at sealed condition for 28 days. For acquiring greyscale based digital material structure, a small cement paste prism with cubic cross-section of 500 µm × 500 µm and length of 2 mm was produced and scanned by a Micro CT-Scanner. The voxel resolution was chosen as 2 × 2 × 2 µm^3/voxel. A cubic region of interest (ROI) with a length of 200 µm was extracted from the specimen for the statistical analysis.

A Continuous Stiffness Method (CSM) developed by Oliver and Pharr [8] was used for the statistical nanoindentation test. 1500 indents were tested and the indentation depth was set as 700 nm. The average modulus and hardness were determined in the displacement range between 400 nm and 660 nm in order to make the interaction volume comparable with the voxel size of XCT images (the interaction volume of nanoindentation is generally around 3–5 h_{max} , where h_{max} is the maximum indentation depth [9]).

The two PDFs were linearly normalized to the range of 0 to 1 with a bin size of 0.01 (Figure 1a). A two-sample Kolmogorov-Smirnov (K-S) [10] test was performed and the results show that the two samples are supposed to be drawn from the same distribution with a 95 % confidence level. An empirical model in a form of power exponent (H_{local}=a$E_{local}{}^{b}$) was proposed to correlate the hardness with its corresponding Young's modulus and shows a good fit with a determination coefficient (R^2) of 0.90 (Figure 1b). The ratio between micro hardness and tensile strength is found to be around 12 [1]. Therefore, the local elastic modulus and tensile strength can be further correlated with the greyscale level. Relationships developed in this section were used in the micromechanical model as further described.



(a)



(b)

Figure 1: Experimental results: (a) comparison of probability density of two distributions; (b) relationship between hardness and Young's modulus from nanoindentation

3. MODELLING

A lattice-type fracture model [11] was used in this work for micromechanical modelling. A volume of cube with length of 100 µm (50 voxels) was randomly extracted from the greyscale images and overlaid on a lattice network. The local micromechanical properties were assigned according to the corresponding greyscale level of the voxel. After mapping micromechanical properties on the lattice mesh, a computational uniaxial tensile test was performed. Nodal displacement was imposed at one side while the deformation of nodes at the opposite side was completely restrained.

The simulated stress-strain curve are compared with the one considering 4 discrete phases in Figure 2. Their corresponding correct patterns are shown in Figure 3. Similar fracture pattern and stress-strain response are found in between the 4-phase method and greyscale level based method. Therefore, it is difficult to determine which method gives more satisfactory results on the micromechanical modelling, but the proposed approach is more generic and direct. It requires less processing steps (no need for deconvolution or averaging of properties, which might introduce errors) and can be always applied once the link is made between the greyscale value and the micromechanical properties.

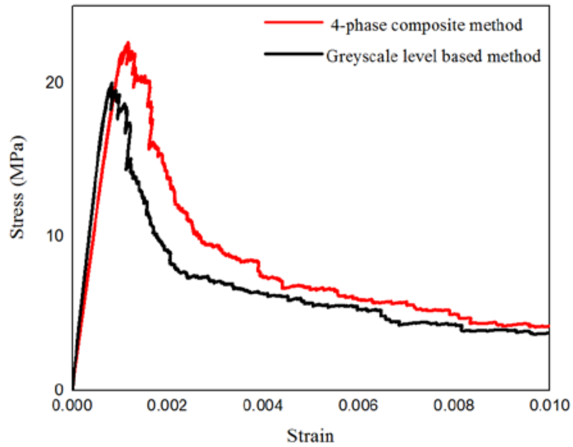


Figure 2: Comparison of simulated stress-strain diagrams for greyscale level based microstructure and 4-phase microstructure

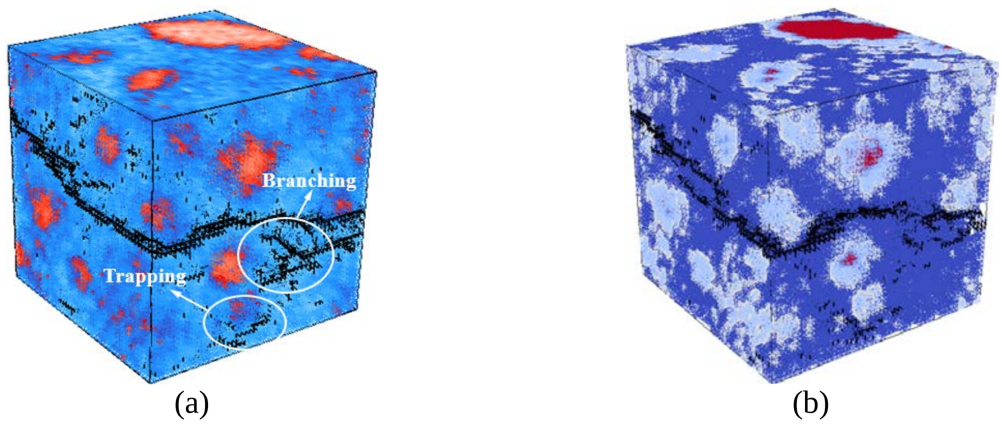


Figure 3: Comparison of simulated fracture pattern of (a) greyscale based microstructure and (b) 4-phase microstructure.

5. CONCLUSIONS

In this work, a new approach for micromechanical simulation of cement paste that combines statistical nanoindentation and XCT technique as input is proposed. The PDF s (i.e. histograms) of Young's modulus and greyscale value were normalized linearly and tested by a two-sample K-S statistics, showing that a strong linear relationship exists between them. The micromechanical properties (E modulus and micro hardness) can be mapped to the voxels according to its greyscale level. The fracture behaviour of greyscale level based microstructure is also compared with the method considering discrete phases. The proposed method is promising, because it captures the gradient of material properties in cement paste that is more realistic. However, a physical understanding behind the relationship between the CT data and local micromechanical properties is still not sufficiently understood and deserves further study.

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