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Testing and modelling of micro cement paste cube under indentation splitting

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ABSTRACT: Cement paste is the glue that holds concrete together and any improvements in material performance will come from its microstructure. For better understanding and predicting its elastic properties and fracture performance, more and more studies are carried out based on micromechanics simulations. However, the predicted results can be hardly verified experimentally due to the technical limitations. This paper presents a procedure for validating micromechanics simulation by making, testing and modelling deformation and fracture of micro cement paste cube (100 μm $\times$ 100 μm $\times$ 100 μm). The micro scale specimens were produced by a micro dicing saw which is commonly employed in the semiconductor industry and fractured by a commercial cylindrical wedge tip mounted on a nano-indenter equipment. A combination of X-ray computed tomography technique and a discrete lattice fracture model was applied to simulate the deformation and fracture performance of the micro scale specimen under indentation. Mechanical properties of local phases are the input for this fracture simulation, which are taken from the previous study by the authors, wherein a micro scale experiment is developed to calibrate these values. Load-displacement curve and crack pattern from the simulation show a good agreement with those obtained experimentally. The proposed technique forms the basis for experimental validation of simulation tools that are used in a multi-scale framework at every scale.

1 INTRODUCTION

As the basic binding material in concrete, cement paste has generated considerable research interest. It is generally accepted that fracture of conventional concrete material starts from micro cracks of cement matrix 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 micro scale is therefore of significant practical importance and scientific interest.

As an effective tool, nanoindentation has been implemented for quantification of local properties like elasticity and hardness of micro level components in the matrix (Constantinides & Ulm 2004, Hu & Li 2014). This technique provides a meaningful experimental input for analytical and numerical models to calculate the global micromechanical properties of cement matrix (Sanahuja, Dormieux, & Chanvillard 2007, Pichler, Hellmich, Eberhardsteiner, Wasserbauer, Termkhajornkit, Barbarulo, & Chanvillard 2013, Luković, Schlangen, & Ye 2015, Zhang & Jivkov 2016) which can be further used as input for a multi-scale framework to simulate the macroscopic mechanical performance of concrete (Pichler & Hellmich 2011, Hlobil, Šmilauer, & Chanvillard 2016, Qian, Schlangen, Ye, & van Breugel 2017, Zhang, Šavija, Figueiredo, & Schlangen 2017). For the past decades, a lot of valuable micromechanical information is obtained to set a basis for understanding and improving the macroscopic mechanical performances. However, for a number of reasons that include problems with producing and measuring miniaturized mechanical samples, these predicted micromechanical properties can be hardly verified experimentally.

In this paper a method on producing and testing micro cement paste cube is reported. The micro scale specimens are produced by a micro dicing saw and fractured by a commercial cylindrical wedge tip mounted on a nano-indenter equipment. In parallel with the experiments, a discrete lattice model is introduced here to simulate the deformation and fracture performance of these micro scale specimens under indentation. The simulated crack pattern and load displacement diagram are then compared with the experimental observations.

2 EXPERIMENTAL

In the experimental program, a grid of micro-cubes ($100 \times 100 \times 100$ μm) was produced using a method developed by the authors (Zhang, Šavija, Chaves Figueiredo, Lukovic, & Schlangen 2016) and tested by the nano-indenter equipment. The method is shortly presented here. 28-day cured cement paste specimens with 0.4 water-to-cement ratio were first glued on top of a glass substrate. The specimen thickness was then made equal to the desired thickness (100 μm), and this was done using a Struers Labopol-5 thin sectioning machine. The micro-cube grid was then fabricated using a precise diamond saw (MicroAce Series 3, Loadpoint, Swindon, UK) which is commonly employed in the semiconductor industry to create silicon wafers. In the machine, a 260 μm thick blade was run in two perpendicular directions over the specimen and the glass substrate (figure 1). The procedure results in a grid of micro-cubes ($100 \times 100 \times 100 \pm 4$ μm) that are used for micromechanical testing(figure 2a).

For testing of the micro-cubes, the nanoindenter is employed. For the purpose of this splitting test, a diamond cylindrical wedge tip (radius 9.6 μm, length 200 μm, see figure 2b) was used in order to apply the load across the middle axis. The experiments were run using displacement control with a loading rate of 50 nm/s up to the failure of micro-cube (figure 2c). Force and displacement data were acquired using the continuous stiffness measurement (CSM) technique (Li & Bhushan 2002) and the applied CSM settings were: 2 nm amplitude, 45 Hz frequency and 100 N/m surface detection.

A typical load-displacement curve recorded by the nano-indenter is shown in figure 3. Clearly two regimes as well as the maximum load point at failure stage can be distinguished from this curve. In regime (I), the load on sample increases monotonically until reaching the maximum load. Once the load exceeds maximum load, the system transitions from a stable regime (I) towards an unstable regime (II). The maximum load can be further used to estimate splitting tensile strength of these micro-cubes (Šavija, Zhang, & Schlangen 2017). The horizontal line in regime (II) indicates an overshoot behaviour of the wedge indenter tip towards the substrate because of the structural collapse of the micro-cube. Since displacement control of the nano-indenter is not fast enough, it is not possible at present to capture the post peak-peak behaviour of the specimen.

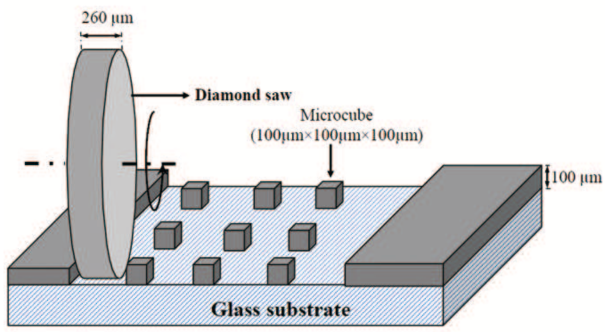

Figure 1: Schematic view of the specimen preparation procedure

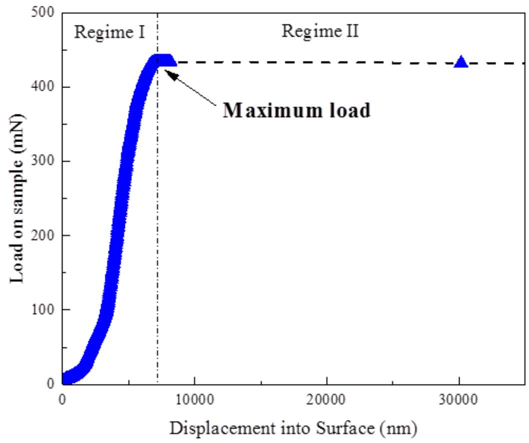

Figure 3: A typical load versus displacement response measured in the micro-cube splitting test.

3 MODELLING

In the modelling part, fracture simulations with a lattice model (Schlangen & Garboczi 1997) were performed on voxel-based cement paste specimens with the same dimension (figure 4). These specimens were generated by X-ray computed tomography (XCT) with a resolution of 2 μm^3/voxel, and consists of four phases including pore (P), anhydrous cement grain (A), inner hydration product (I) and outer hydration product (O). More information about the experiments, reconstruction and image segmentation procedure can be found in (Zhang, Šavija, Chaves Figueiredo, Lukovic, & Schlangen 2016).

In the lattice model, the material is assembled by a set of beam elements having linear elastic behaviour. Then, a set of linear elastic analyses is performed by calculating the nodal responses of the lattice network for an external boundary displacement. At every analysis step, a unit displacement is applied, a critical beam element with the highest stress/strength ratio is labelled and removed from the mesh, thereby introducing a small crack. This procedure is then repeated with the updated geometry and stiffness of the whole lattice network until structural failure happens. As a consequence, the fracture pattern of the investigated material volume at each step can be obtained as well as their load-displacement response which can be further converted to the laboratory observed load-displacement diagram of the specimen under loading.

As shown in figure 5, the voxel-based specimen

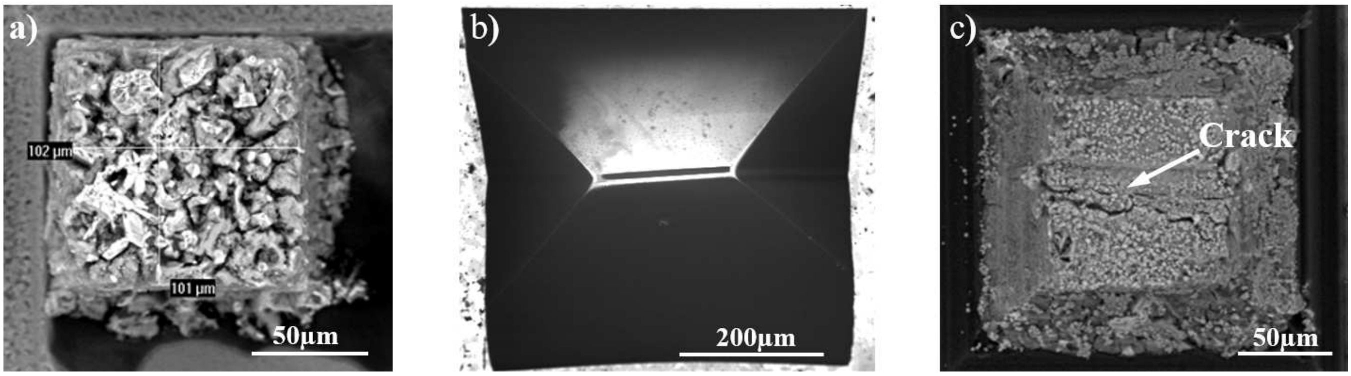

Figure 2: Environmental scanning electron microscope image of (a) small cement paste cube on glass plate (b) cylindrical wedge indenter tip and (c) damaged cement paste cube.

is discretized as a lattice mesh. The microstructure of the material can be mapped onto these beam elements by assigning them different properties, depending on the phase type of connected two voxels by the beam element. Three solid phases in the microstructure result in six types of lattice elements. Elasticity modulus of beam element was ascribed with the harmonic average of the connected two phases, while the strength assigned as the lower value in between. The mechanical parameters of each single phase used in this study are presented in table1. Elastic moduli are assumed equal to the nanoindentation measurements for individual phases (Hu & Li 2014). The tensile strengths are taken from a previous study by the authors (Zhang, Šavija, Chaves Figueiredo, Lukovic, & Schlangen 2016), wherein a micro scale experiment is developed to calibrate these values. The compressive strength of each phase is estimated 20 times as high as its tensile strength. The lattice elements (coloured grey in figure 5) located below 20 μm from the bottom were set as glue element with a low elastic modulus of 3 GPa (measured by a grid nano-indentation test) to represent the glue layer between cement paste and glass substrate. These elements were not allowed to fail in the simulation (i,e. they remain linear elastic). For the boundary conditions in the simulation it was assumed that the displacement of the nodes at bottom surface was fixed in all directions to represent the glued sample on the glass plate. A vertical displacement was applied on nodes in the two lines closed to the middle axis of the top surface to mimic the indenter load.

The damaged specimen and crack pattern are presented in figure 6 and figure 7 respectively. It can be clearly seen that the simulated fracture pattern is comparable to the experimental observation. A lot of damage occurs under the loading points, and the final crack pattern results in one main crack under the indenter. The simulated load-displacement curve is plotted in figure 8 together with several experientially measured curves. As the post-peak behaviour of specimen cannot be captured due to current technical limitation, validation of the numerical model was restricted only in regime (I). The simulated load-

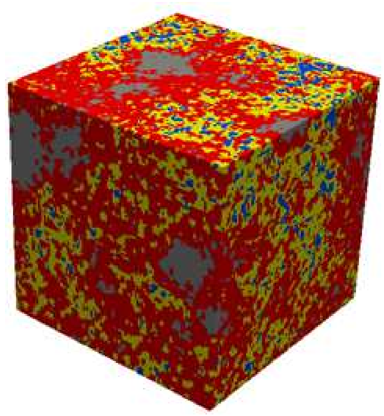
Figure 4: Microstructure of cement paste with size 100 μm $\times$ 100 μm $\times$ 100 μm at the curing age 28 days and 0.4 water-to-cement ratio from XCT experiments (grey-anhydrous cement; red-inner product; yellow-outer product; blue-pore).

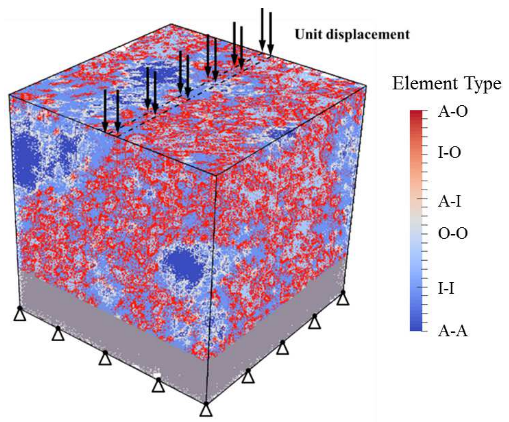

Figure 5: Computational splitting test on lattice mesh of micro cement paste cube.

displacement curve and one of the experimentally measured cures show a high degree of consistency on the peak load and stiffness (slope of the load displacement curve). Due to the fact that some slip occurs at the beginning of the experiments, the measurements are slightly shifted, but the slope remains similar to the one in simulated load displacement curve. It is observed that the test results still show a high variability which is induced by the inherent heterogeneity of this material. Therefore, it is suggested that in future investigation, multiple voxel-based specimens need to be generated and tested to obtain statistical results on the micromechanical properties.

Table 1: Assigned local mechanical properties of individual phases.

Phase	Modulus(GPa)	Tensile strength(GPa)	Compressive strength (GPa)
Pore	0.0	0.00	0.00
Anhydrous cement	99.2	0.68	13.2
Inner product	31.2	0.09	1.8
Outer product	25.2	0.06	1.2

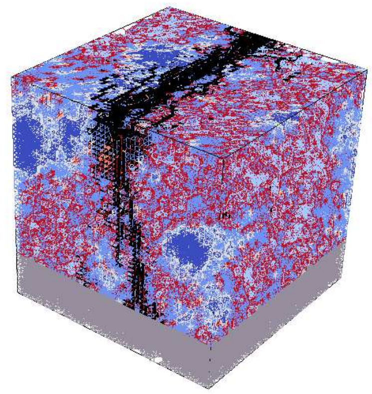

Figure 6: Damaged specimen in the final failure state under indentation splitting (black-crack).

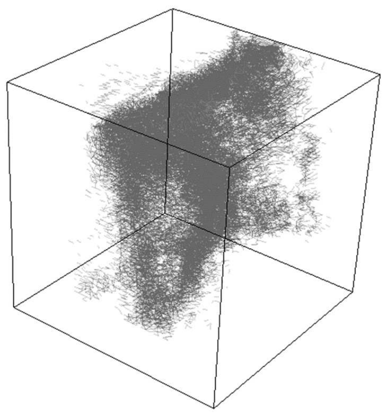

Figure 7: Simulated crack pattern in the final failure state.

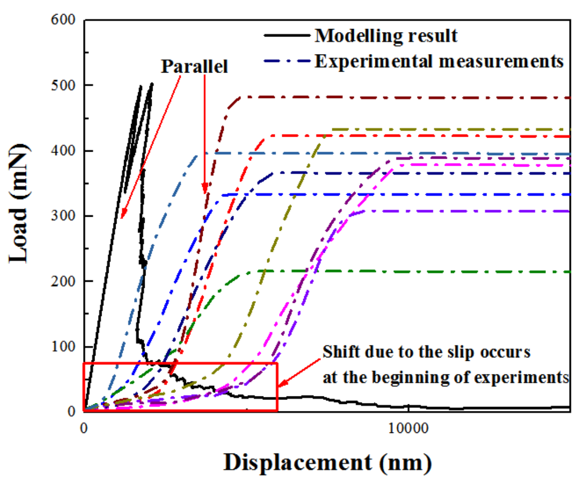

Figure 8: Comparison between simulated load displacement diagrams and experimental results.

4 CONCLUSIONS

In this paper the failure mechanism in cement paste specimens at micro scale was studied by a combination of experimental technique using nano-indenter equipment and a modelling technique using discrete lattice model. The input for the simulation are mechanical properties of the individual local phases, which are calibrated from the previous study by the authors, wherein a micro scale experiment is developed to calibrate these values. The simulated fracture pattern and load-displacement curve are compared with the experimental observations of same size specimens. It is showed that these input local mechanical properties can be applied to fracture simulations under different boundary conditions and have satisfactory results. With the method presented in this paper the framework for validation of the modelling results at micro scale is created. This method forms also a basis for validation of multi-scale modelling results at every scale.

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