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TESTING AND MODELLING OF FIBRE-MATRIX INTERFACE BY MICROCUBE SPLITTING

YANCHEN HE[1,2], SHAN HE[1,3], ERIK SCHLANGEN[1]

[1] Delft University of Technology, Faculty of Civil Engineering and Geosciences, Microlab, Stevinweg 1, Delft, The Netherlands
e-mail: erik.schlangen@tudelft.nl, www.tudelft.nl

[2] Hong Kong University of Science and Technology (HKUST), Hong Kong
e-mail: yhedm@connect.ust.hk

[3] Witteveen & Bos, The Netherlands

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Abstract: This study examines the impact of microfibre inclusion on the tensile properties of cement paste at the microscale through both experimental and simulation approaches. Micro-cubes containing vertically aligned microfibres were fabricated using cement pastes with varying water-to-cement (w/c) ratios, ranging from 0.3 to 0.5. These specimens underwent a splitting test using a nano-indenter with a wedge tip, and the results were compared to those of micro-cubes without fibres. Mechanical properties, including load capacity, peak deformation, stiffness, and fracture energy, were analysed. The findings indicate that fibre inclusion reduced the splitting tensile stress and modulus of the micro-cubes across all w/c ratios. Additionally, the influence of fibre inclusion became more pronounced with higher w/c ratios, likely due to a more porous interfacial transition zone (ITZ) at these higher ratios. To complement the experimental work, a lattice model with a simplified microstructure, comprising paste, fibre, and ITZ, was developed to simulate the fracture behaviour of the micro-cube under indentation splitting. The simulation results were in strong agreement with the experimental data, facilitating the determination of the mechanical properties of the ITZ.

1 INTRODUCTION

The Interfacial Transition Zone (ITZ) in fibre-reinforced concrete (FRC) refers to the microscopic region surrounding the microfibres within the cement-based matrix, which exhibits a more porous microstructure than the bulk matrix. This porosity has led to the common belief that the ITZ serves as a weak link between the constituents. Since the efficiency of force transfer between the fibre and the matrix plays a critical role in the overall performance of FRC structures, the properties of the ITZ are essential in determining the mechanical properties of the composite material [1].

In the tensile testing of FRC, the initial cracking strength is governed by the matrix strength, while the post-cracking behaviour is primarily influenced by the bond between the fibre and the matrix [2]. To investigate the bond-slip behaviour and understand the bonding mechanism, a single fibre pull-out test has been developed. This test measures the force required to extract a monofilament fibre from its matrix by conducting a tensile test on the partially embedded material [2,3]. The evaluation of the pull-out process is based on two key parameters: the maximum debonding load and the debonding energy, both of which are strongly influenced by the tensile behaviour of the ITZ. Although much research has focused on characterizing the microstructure of the ITZ or measuring its indentation modulus, the tensile properties of the ITZ have not been thoroughly explored. These properties are crucial in controlling the bond behaviour between the fibre and the cement matrix, highlighting a significant gap in the current research.

To address this gap, a micro-splitting tensile test was conducted to examine the tensile behaviour of the ITZ. This test involved applying a line force at the centre of the top surface of microcubes containing a single fibre, using a wedge tip mounted on a nano-indenter. In addition to the experiments, a lattice model with a simplified microstructure was developed to evaluate the mechanical properties of the ITZ and simulate the fracture behaviour of the tested micro-cubes [4].

2 EXPERIMENTS

2.1 Materials and methods

Cement paste specimens with varying w/c ratios of 0.3, 0.4, and 0.5 were produced using CEM I 42.5 N Portland cement. The microfibre used to produce the ITZ is a PVA (Polyvinyl alcohol) microfibre from Kuraray (Japan). The nominal diameter of the fibre is 39 µm. At first, a prismatic mould was fitted with five 150 mm long PVA fibres spaced equally apart. Cement paste was mixed, poured and the moulds were vibrated. The specimens were cured for 28 days (20oC and 98% RH) after which hydration was stopped using isopropanol.

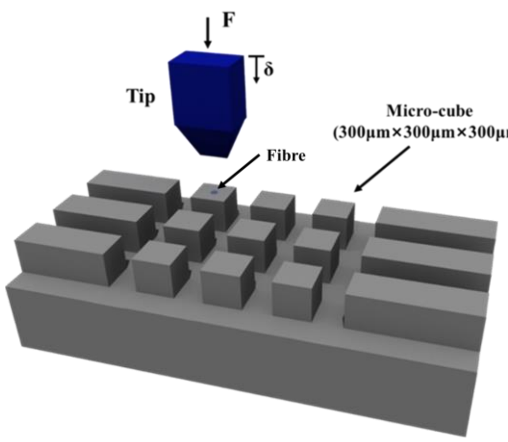


Figure 1: Schematic illustration of micro-cubes and loading with wedge-tip.

Afterwards, the cement paste prism embedded with fibres was sliced by a diamond saw. MicroAce Series 3 Dicing Saw was used to fabricate the microcubes from the slices. Always a 3 by 3 microcube matrix was produced, consisting of one cube with fibre in the upper left corner and remaining eight cubes without, see figure 1. The micro-cubes were designed with a dimension of 300 µm.

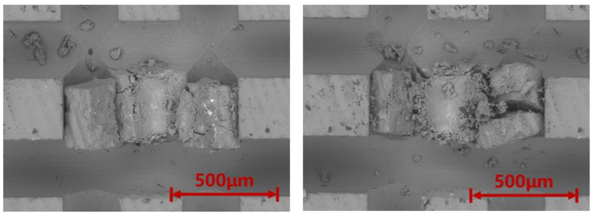
An Agilent G200 Nano Indenter equipped with a diamond wedge tip was used to conduct the microcube splitting test. The length and radius of the cylindrical edge are 200 µm and 9.6 µm, respectively. A line load is applied at the centre of the top surface, with a constant displacement increment of 50 nm/s until the cube fails.

2.2 Results

Compared to conventional nano-indentation tests with Berkovich indenters, results obtained from the indentation splitting test are more representative of the global mechanical performance of micro-cubes, for the crack typically propagates through the ITZ due to its porous nature. Given that the PVA fibre-matrix interface size generally ranges from 100-150 µm [1].

Figure 2 shows the SE image of the failed specimens with and without fibre. Since the maximum tensile stress is always present in the central axis below the tip, it is clear that the specimen without fibres is divided into left and right sections. Except for the cement paste, the fibre was also splitting into two halves by the indenter tip. For the specimen with fibres, there is a noticeable secondary crack on the right side in addition to the primary crack in the middle. ITZ has a larger porosity than cement paste, and owing to its irregular pore distribution, it is susceptible to stress concentration at specific high porosity regions. Following the formation of the main crack, the secondary crack develops from ITZ close to the edge of the fibre notch.

Representative load-displacement curves of specimens with fibre and without fibre with a w/c ratio of 0.4 are shown in Figure 3. The load on the sample increases monotonically until it reaches the critical splitting load (maximum load). After the maximum load, the wedge indenter tip had an overshoot due to the structural collapse of the micro-cube, as indicated by the horizontal line. This cannot be controlled by the nanoindenter system.



a) b)

Figure 2: SE image of failed specimen: (a) Reference, (b) Fibre.

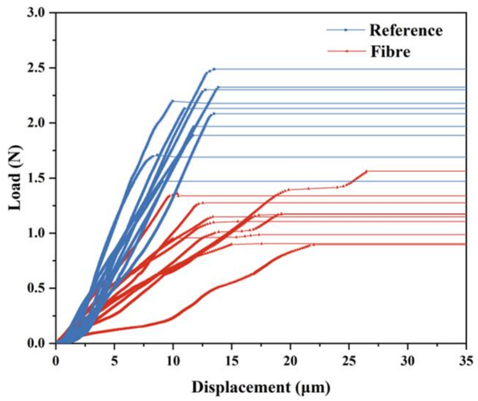


Figure 3: Load-displacement response with and without fibre under w/c ratio of 0.4.

The figure shows that the specimen without fibre has a peak force between 1.5 and 2.5 MPa, which is higher than the specimen with fibres (between 0.75 and 1.5 MPa). In addition, the displacement of fibre specimens is higher than that of the reference group, suggesting that the presence of fibres reduces the stiffness of the microprism.

Figure 4 displays the splitting tensile strength results for the microcubes with and without fibres under various w/c ratio. As expected, an increase in the w/c ratio results in a decrease in the splitting tensile strength for both fibre-reinforced and unreinforced experimental groups. Moreover, at each w/c ratio, the strength of specimens with fibres is significantly lower than that of specimens without fibres.

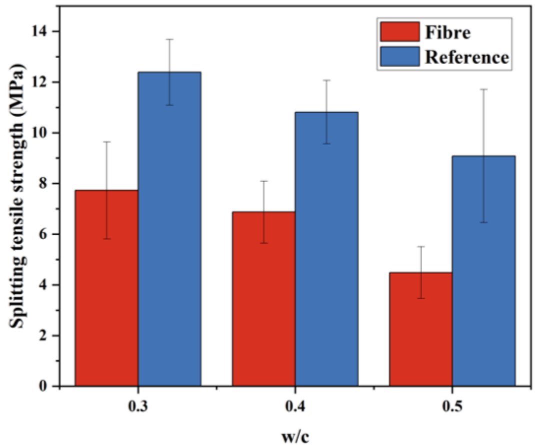


Figure 4: Splitting tensile strength of microcubes

3 SIMULATIONS

3.1 Model and assumptions

A 3D lattice model [5] was used to simulate the stress-strain response, cracks pattern and microcracks propagation in the microcubes. The mechanical properties of ITZ that were obtained could serve as a reference for the larger-scale simulation.

The 3D mesh was generated as shown in figure 5. To control computational cost, the size of the model was set as 30×30×30, and the cubic domain was divided into a cubic grid with a cell edge length of 10 µm. the nodes on the bottom surface were clamped to represent the connections between the microcubes and their substrate. A vertical displacement was applied to the nodes in the centre top of the specimen, covering a width of 10 µm and a depth of 30 µm, to simulate the indenter load. The failure mode of the boundary condition nodes was set to neither tension nor compression to avoid affecting the experimental results.

The diameter of the fibre is 39 µm, and the outer diameter of the ITZ is set as 100 µm according to the observation in the previous study [1]. The schematic view of the simplified model is shown in figure 6. The reference model consists of a single phase, cement, while the fibre model includes three phases: fibre, ITZ, and paste.

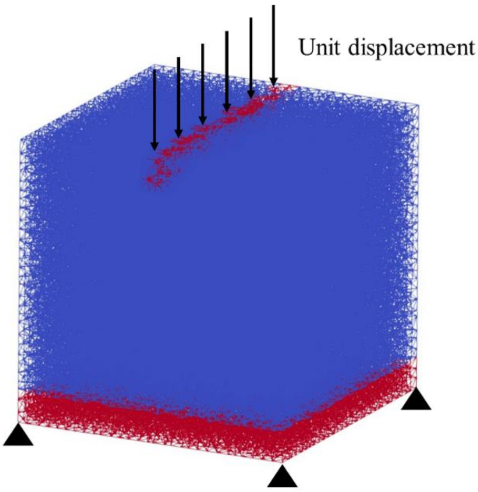


Figure 5: Schematic view of mesh and boundary conditions

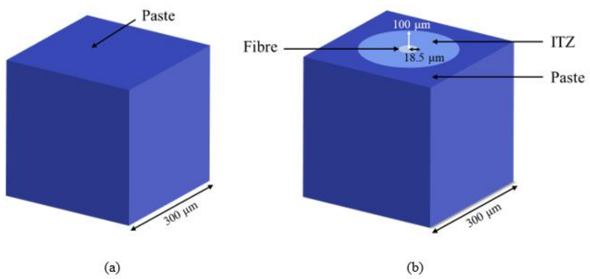


Figure 6: Schematic view of (a) reference model; (b) fibre model.

The properties of the elements are taken according to the values given in table 1. The strength and stiffness of the paste is chosen such that the slope and the peak load of the simulations match with the experimental outcome. In the simulation of the specimen containing the fibre the stiffness and strength of the elements in the ITZ-zone is taken as a certain percentage (20-80%) of the strength and stiffness of the elements in the paste-zone.

Table 1: Local mechanical properties of lattice elements

Phase		Young's modulus (GPa)	Compressive strength (MPa)	Tensile strength (MPa)
Fibre		12.5	50	50
Paste	w/c 0.3	22	280	22
	w/c 0.4	20	200	19
	w/c 0.5	16	140	16

3.2 Results

Figure 7 presents a comparison between the simulated load-displacement curves of fibre specimen models with w/c ratios of 0.4 and the reduced values for the ITZ properties. The experimental load-displacement curves of 10 specimens were plotted for comparison. The experimental results exhibit a large range of variability, so a red dashed line representing the average experimental results is included to facilitate comparison with the numerical results at different material parameter inputs.

For all w/c ratios, the range of the experimental results encompasses the 20% and 40% numerical simulation results, with the average experimental result being closer to the 20% material input simulation. This provides a reference for the material input parameters considering the ITZ in the simulations, suggesting that regardless of the w/c ratio, the material properties of the ITZ are approximately equivalent to 20% of the paste properties.

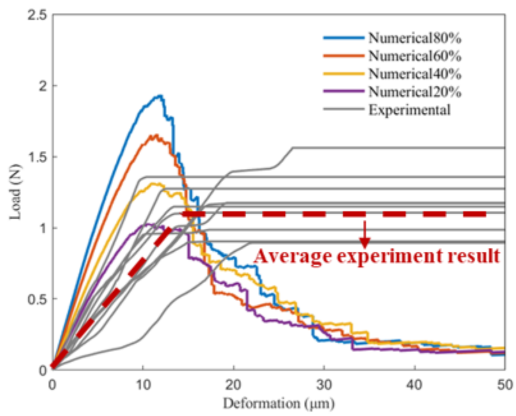


Figure 7: The experimental and numerical results of homogeneous fibre specimen models with w/c of 0.4.

Figure 8 presents the simulated crack pattern of a micro-cube containing fibre under indentation splitting. The model includes five types of elements. The red-highlighted elements in the figure indicate failed elements, with subfigures (c), (f), and (j) marking the types (paste, ITZ, fibre) of these failed elements. The figure only shows the crack development for the w/c ratio of 0.3, as the simulated fracture behaviour for the other two w/c ratios is similar to that of 0.3.

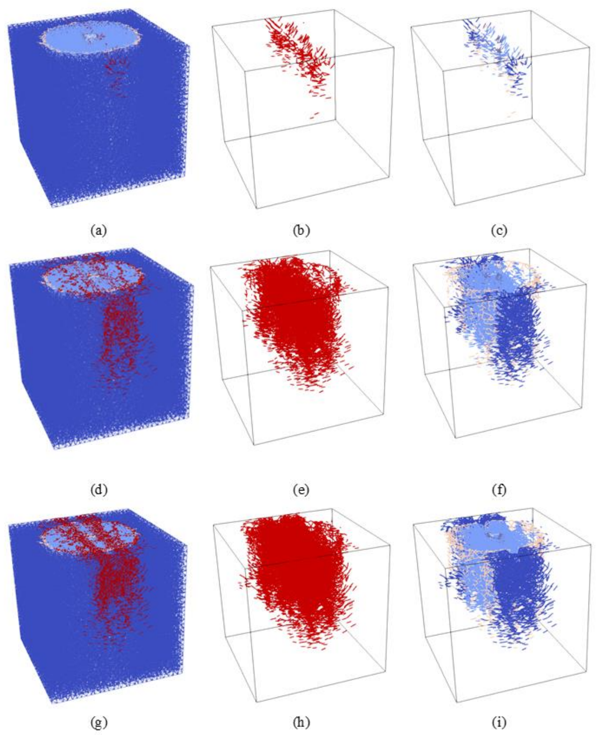


Figure 8: Simulated crack pattern of a micro-cube containing fibre under indentation splitting: (a) crack initiates at the centre of the upper part of the specimen; (b) visualization of the crack at the initial stage; (c) visualization of failed element at the initial stage; (d) crack propagates from the middle part of the specimen; (e) visualization of the crack at the crack propagation stage; (f) visualization of failed element at the crack propagation stage; (g) crack propagates through the centre of the micro-cube and it is split into two halves; (h) visualization of the crack at the final stage; (i) visualization of the failed elements at the final stage.

4 DISCUSSION AND CONCLUSIONS

This study developed and simulated a framework for fabricating microcubes embedded with a single microfibre to investigate the fibre-matrix interfacial transition zone (ITZ). A lattice model with a simplified microstructure was used to explore the tensile properties of the ITZ. The splitting tensile behaviour was assessed using a nanoindenter with a diamond wedge tip. The impact of fibre inclusion on the mechanical properties of microcubes was examined across various water-to-cement (w/c) ratios. Additionally, the fracture behaviour of microcubes under indentation splitting was simulated using a microstructure-informed lattice fracture model, based on experimental observations.

The experimental results confirmed that the proposed method for fabricating microcubes maintained specimen integrity, and the indentation splitting test proved effective for evaluating the tensile behaviour of the fibre-matrix interface. Simulation results closely matched experimental data, except for post-peak behaviour, which could not be captured experimentally due to instrument limitations.

The mechanical properties of the local phases were calibrated by comparing the simulated results with the experimental data, revealing that these properties are crucial for understanding load-displacement responses and failure mechanisms. The modelling indicated that the material properties of the ITZ are approximately 20% of the properties of the paste, regardless of the w/c ratio.

More detailed results of both experiments and simulations can be found in [6].

REFERENCES

[1] S. He, Y. Chen, M. Liang, E.-H. Yang, E. Schlangen. 2023. Distribution of porosity surrounding a microfiber in cement paste, *Cement and Concrete Composites* **142**.

[2] C. Lin, T. Kanstad, S. Jacobsen, G. Ji, 2023. Bonding property between fiber and cementitious matrix: A critical review, *Construction and Building Materials* **378**.

[3] F. Teklal, A. Djebbar, S. Allaoui, G. Hivet, Y. Joliff, B. Kacimi. 2018. A review of analytical models to describe pull-out behavior - Fiber/matrix adhesion, *Composite Structures* **201**: 791-815.

[4] Y. He. 2024. Testing and Modelling of the Mechanical Properties of Fibre-matrix Interfacial Transition Zone. *MSc-thesis*. Delft University of Technology.

[5] H. Zhang, Y. Xu, Y. Gan, Z. Chang, E. Schlangen, B. Šavija. 2019. Combined experimental and numerical study of uniaxial compression failure of hardened cement paste at micrometre length scale, *Cement and Concrete Research* **126**.

[6] Y. He, S. He, M. Liang, E. Schlangen. 2025. Indentation splitting test of cement paste micro-cubes containing single microfibre. *Submitted for publication.*