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Indentation tests based multi-scale random media modeling of concrete

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HIGHLIGHTS

- Systematic nanoindentation tests performed on each constituent of concrete at two scales.
- Random medium modeling of concrete with a novel reconstruction technique.
- Local averaging modeling between the nano- and micro-scales.

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ABSTRACT

In the present paper, a multi-scale random media model is proposed based on the indentation tests for each constituent of concrete including hydrated cement paste (HCP), aggregate and interfacial transition zone (ITZ). Firstly, systematic indentation tests are performed for each constituent of concrete at the nano- and micro-scales. Following the random field theory, each constituent of concrete materials is modeled as a random field. The scale of fluctuation is investigated based on the results of indentation tests. At the nano-scale, the scales of fluctuation of HCP and ITZ both turn to be roughly 20 μm, but that of aggregate is much larger. At the micro-scale, the scales of fluctuation of HCP and aggregate stay from 167 to 569 μm. Then the pointwise parameter estimation and model verification are performed for each constituent of concrete, and the one-dimensional (1-D) probabilistic density function (PDF) of the random field is obtained based on the proposed maximum possibility criterion. The probability distributions of the indentation modulus and hardness for each constituent of concrete are identified based on the statistical analysis. By introducing the reconstruction technique, concrete materials could be reconstructed as the random medium at the nano- and micro-scales. With the local averaging theory, the reduce factor for mechanical properties between the nano- and micro-scales is studied and it is shown that the experimental reduce factor agrees well with the theoretical one.

1. Introduction

It is generally believed that the properties such as elastic modulus, strength and fracture energy, are governed by the nanoscale properties [1], which are not intriguing with the development of indentation techniques. In recent years, indentation tests have been widely used to investigate the properties for cementitious materials. In early years, the microhardness tests were used to study the bulk properties of cement paste with a maximum penetration depth in the level of 10^{-5} m [2,3]. With the significant advances for the conventional Vickers microhardness testing technique, a novel microindentation technique was developed to investigate the elastic modulus and the microstrength of ITZ in reinforced concrete [4–6]. To obtain the submicron-scale mechanical properties of cement paste, the nanoindentation technique was emerged and applied to determine the first indentation results of individual constituents of Portland cement clinker with the penetration depth around 0.3–0.5 10^{-6} m [7]. With the help of nanoindentation technique, the existence of two types of calcium–silicate–hydrates (C-S-H) with the corresponding volume fraction was found in Refs. [8,9]. Based on the work of Refs. [8,9], a statistical analysis of massive nanoindentation tests, namely the grid indentation technique, was proposed to allow for the information on the phase mechanical properties and the volume fraction [10,11]. Later, the previous work was extended to the bone and the shale [12], C-S-H at elevated temperatures [13], the ultra-high performance concrete [14], and so on. As to ITZ, several works have been done to provide better insight into the mechanical properties in cement paste [15], steel fiber reinforced mortar [16], and recycled aggregate concrete [17].

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The statistical information (e.g. the PDF and the relative volume fraction) of cement paste can be provided by the previous work. However, in the field of structural engineering, ones always pay more attention to the probabilistic and statistical knowledge about concrete instead of mortar and cement paste. Inspired by the previous researches, a statistical way is proposed in the present paper to model concrete at the nano- and micro-scales.

This study is organized as follows. Section 2 presents the experimental details for the nano- and micro-indentations. In Section 3, we conduct the probabilistic and statistical analysis on each constituent of concrete, e.g. HCP, ITZ and aggregate, and obtain the corresponding 1-D PDF of the random field by a "maximum possibility criterion". After that, a random media model of concrete is proposed by synthesizing the PDFs of the three constituents in Section 4. Section 5 develops the intrinsic connection between the nano- and micro-scales using the local averaging theory. Numbers of conclusions are given in Section 6.

2. Materials and methods

The material prepared herein was the regular concrete, which was casted into steel molds at the water: cement: sand: aggregate ratio of 0.4:1:2:5, to form bars with the dimensions of 0.1 m × 0.1 m × 0.3 m. The specimens were hydrated with a humidity of 95% at the room temperature and kept in the standard conditions for 3 months.

The specimens were then cut into several small sample blocks with the size of 20 mm × 20 mm × 20 mm. To meet the requirements of the nano- and micro-testing, small specimens with the approximate dimensions of 20 mm × 20 mm × 5 mm were made up using a diamond saw. The small samples were embedded into the epoxy resin firstly. In the second step, according to the previous polishing technique [15,17], the small specimens were ground and polished with silicon carbide papers down to 6 μm, then the diamond lapping films of gradations 6, 3, 1, and 0.5 μm were used to polish the samples. To avoid the effect of surface roughness on the indentation results, the measured root-mean-square roughness of the sample surface should be lower than one third of the average indentation depth [18]. Finally, the small samples were ultrasonically cleaned in water for 1 min, in order to remove the polishing debris.

The NanoTest Vantage was used to measure the elastic properties of materials at the nano- and micro-scales, by using electromagnetic force application and capacitive depth measurement. A Berkovich tip was used for the indentation. According to Oliver and Pharr method, the mechanical properties where the indentation hardness is defined as the maximum load divided by the contact area and the modulus is obtained from the final unloading curve can be obtained from the loading-unloading curves [19]. The indentation law for the nano- and micro-indentations was shown in Table 1. A series of nano- and micro-tests were conducted on the specimens shown in Fig. 1. To acquire the properties of each constituent of concrete, the preselecting testing zones were made on the specimens with regard to each constituent. For the simplicity, the aggregate was abbreviated as the capital letter "A", HCP as "H" and ITZ as "I". Each indentation area and its corresponding indentation lattice were listed in Table 2. For the tests at the nanoscale, the dimensions of the indent lattice for HCP, ITZ and aggregate were all 25 × 20. And for the micro-testing, the indent lattice dimensions for HCP and aggregate were both 14 × 20.

Table 1
Indentation law.

Control factor	Nano-test	Micro-test
Maximum depth	300 nm	10 μm
Loading and unloading rate	0.2 mN/s	100 mN/s
Hold time	15 s	15 s
Grid space	seeing Table 2 and Fig. 2	

Taking the indent areas $A_{n,1}$ and $A_{m,1}$ for instance, the schematic diagrams are shown in Fig. 2.

For a relatively large indentation depth, due to the restrain of the stress field and the physical interaction of the penetrating indenter and the rigid inclusion, the penetration near a rigid inclusion surface would be reduced [20]. In addition, as for the micro-tests, the indentations operated to a depth of h_{max}, would activate material situated on a surface radius of $4h_{max}$ [21], which may be larger than the width of ITZ. To avoid this argument, the indents on the ITZ phase were not considered in the tests at the microscale, as detailed in Table 2.

3. Probabilistic analysis on each constituent

3.1. Probabilistic homogenization

In reality, concrete with its attribute displaying sufficient large degree of disorder should be described by a probabilistic rather than the deterministic model. Particularly, the random field theory is born to model the complex distributed disordered system [22]. In the following, each constituent of concrete is treated as a random field.

To derive stochastic model, each sample (random series) with 20 observations from the nano- and micro-tests is divided into 4 sub-samples consisted of 6 observations (seeing Fig. 3). Totally, the sample number for HCP is 100, that for ITZ is 100, and that for aggregate is 100 at the nanoscale, and that for HCP and aggregate both are 84 at the microscale.

At the nanoscale, Fig. 4 displays the sample data, the related first-order and second-order statistics of the nano-hardness for HCP. At the microscale, the statistics of the micro-hardness for HCP are shown in Fig. 5.

Figs. 4a and 5a confirm that most of the values fall within the range from the mean value minus the standard deviation (denoted by "Mean − SD") to "Mean + SD". As shown in Figs. 4a, b and 5a, b, the mean values, the standard deviations and the autocorrelation results for the nano- and micro-hardness remain constant with the variation of space. Moreover, the probability characteristics only depend on the relative locations of the points (the relative distance here τ = 0, 4, 8 and 12 μm at the nano-scale, and τ = 0, 0.2, 0.4, and 0.6 mm at the micro-scale). From the properties listed above, the 1-D random fields for the nano- and micro-hardness of HCP could be considered to be homogeneous. With regard to the nano- and micro-modulus for HCP and the nano- and micro-properties (indentation hardness and modulus) for aggregate and ITZ, the same probabilistic analysis repeatedly applied to the relative random field, it turns out that all the random field could be called homogeneous (stationary). Although the intrinsic complexity in the microstructure and the physical and mechanical properties has not been well understood yet, the concrete constituents can also be regarded as a kind of probabilistic homogenous materials in the current stage of investigation.

3.2. Scale of fluctuation θ

To focus on the scale of fluctuation, consider the correlation function expressed as Eq. (1), based on Vanmarcke' work [22], the scale of fluctuation θ can be derived as Eq. (2).

$$\rho(\tau) = \exp(-a\tau) \tag{1}$$