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Review

A review on the mechanical properties of cement-based materials measured by nanoindentation

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HIGHLIGHTS

- The application of nanoindentation to cement-based materials is reviewed.
- The microstructural mechanical properties of cement-based materials are reviewed.
- The critical aspects in this field are reviewed and discussed.

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ABSTRACT

A well-known fact is that the mechanical properties of cement-based materials largely depend on the material structure at the micro- and nano-scales. To understand and improve the macroscopic mechanical performances of cement-based materials, it is essential to investigate the mechanical properties of cement-based materials at the micro- and nano-scales. Benefited from the application of the nanoindentation technique, a lot of valuable microstructural mechanical information of cement-based materials has been reported during the last decade. As the application of nanoindentation gains more and more interests from the researchers in the field of construction and building materials, this paper aims to provide an overview on this important technique in application to cement-based materials, and present a comprehensive review on the mechanical properties of cement-based materials measured by nanoindentation. In the review, the nanoindentation methods that had been used and the findings that had been reported were summarized. Meanwhile, the critical aspects of nanoindentation in application to cement-based materials were discussed.

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1. Introduction

Nanoindentation is an effective and powerful tool to detect the local elastic properties and hardness of the material microstructure and nanostructure. The basic idea of nanoindentation is simple: push a very sharp tip into the surface of a material and investigate the mechanical behavior of the material from the response of the tip. The pioneering work can be traced back to the work of Brinell, who pushed a small ball to the surface of materials and measured the plastic properties from ball bearings as indenters. In the present, the evolution of nanoindentation technique allows to directly perform such mechanical tests on the microstructure and nanostructure of materials. Since nanoindentation was introduced to study the cement-based materials, a lot of valuable microstructural mechanical information of cement-based materials has been reported. During the last decade, nanoindentation has been used to study the microstructural mechanical properties (e.g., indentation modulus *M*, hardness *H*, and creep compliance *C*) of cement pastes at different water to cement ratios [4,6–12,15,16,23–26,29–31,33,34,37,43,44,46–48,51,56,57,69,72,81,87–89,91,93,97,99,100,102,109,110]. Moreover, nanoindentation has been extended to characterize the microstructural mechanical properties of blended cement pastes by nanosilica [20,37,38,53,111], silica fume [27,75], fly ash [18,27,75,94,95], slag [18,45,94,95] and metakaolin [18,28], as well as alkali-activated cementitious materials such as fly ash [58–60], slag [72] and metakaolin [59], and other cement-based materials [34,76,77,79,103,108]. Meanwhile, the mechanical properties of interface transition zone (ITZ) in aggregate and fiber reinforced cementitious composites were investigated through the application of nanoindentation [1,41,51,52,54,55,61,80,84,96,104–107]. The application of nanoindentation provided an insight into the microstructural mechanical properties of cement-based materials, and a lot of valuable microstructural mechanical information was obtained to set a basis for understanding and improving the macroscopic mechanical performances. Starting from the microstructural information obtained from nanoindentation, a lot of multi-scale analytical and numerical models were proposed and successfully used to calculate the macroscopic mechanical performances of cement-based materials, e.g., [2,8,26,32]. Besides, the microstructural mechanical information of cement-based materials obtained from nanoindentation provided one of the important evidences to the molecular simulation, e.g., [19,63,67,73,78].

Nowadays nanoindentation is becoming a general technique in characterizing the mechanical properties of the existing cement-based materials, and can be potentially used to quantitatively evaluate the microstructural modification in the mechanical properties of the newly developed cement-based materials. Considering the application of nanoindentation gains more and more interests from the researchers in the field of construction and building materials, this paper aims to provide an overview on this important technique in application to cement-based materials, and present a comprehensive review on the mechanical properties of cement-based materials measured by nanoindentation.

2. Sample preparation

Before the nanoindentation tests, the most important is the sample preparation. As the classical nanoindentation analysis is based on the assumption of perfectly smooth surface of material [13,42], the sample preparation is very important in obtaining the reliable and repeatable nanoindentation data. To evaluate the effectiveness of the sample surface preparation, the surface roughness was generally measured by atomic force microscope (AFM) and described by the root mean square roughness number (*RMS*):

$$RMS = \sqrt{\frac{1}{n \cdot m}\sum_{i=1}^{n}\sum_{j=1}^{m}(h_{ij} - h_{mean})^2} \tag{1}$$

where n, m denote the pixel size of the AFM image, h_{ij} is the height reading in pixel (i, j) and h_{mean} is the mean value of all height readings in the image. However, the measurement of *RMS* roughness is highly dependent on the scanning size and the scanning area. To achieve an acceptably smooth surface, the sample preparation procedure generally included the cutting and polishing. According to the previous successful sample preparation, the general procedure was briefly summarized below: firstly, the specimen with small size was cut out and the surface layer of the specimen was removed; Then, the specimen was carefully polished with abrasive papers, diamond suspensions or others in order from rough to fine; Finally, the polished specimen was cleaned to remove debris and suspensions left on the surface. As well, it was noted that focused ion beam milling as a promising surface preparation technique was successfully used in the sample preparation of atomic force microscope indentation [86].

To examine the goodness of the surface condition, Miller et al. [48] proposed a surface criterion of nanoindentation on cement paste based on the results of the statistical nanoindentation technique on a w/c (water to cement ratio) = 0.2 cement paste. In the study of Miller et al. [48], it was demonstrated that the material properties obtained from statistical nanoindentation technique converge to a unique set of values when the average indentation depth h is greater than 5 times the *RMS* roughness where the roughness is measured over a scanning size 200 times h. However, the reliability of statistical nanoindentation technique used in their work was questioned (see 3.3), as well as the surface roughness reported in their work. In the work of Trtik et al. [85], it was indicated that the unaffected surface roughness cannot satisfy the surface criterion of Miller et al. [48] due to the presence of the pores. In the work of Modal [49], it was also noted that a w/c = 0.5 cement paste could be polished to achieve an average *RMS* roughness of 150 nm over an area of $40 \times 40\ \mu m^2$, while it is still much higher than the *RMS* roughness criterion (no more than 40 nm over an area of $40 \times 40\ \mu m^2$) of Miller et al. [48].

It is generally regarded that the indelible surface roughness of cement-based materials is mainly caused by the incorporated pores at the micro- and nano-scales [85], and thus if the nanoindentation data corresponding to the large pore (for the pores that do not satisfy the scale separation, see 4.3) could be got rid of, the more reliable nanoindentation results can be obtained [24,25]. The possible way to identify such area is to couple nanoindentation with the other microstructure/phase characterization techniques like scanning electron microscope and atomic force microscope (see 3.4) [24,25].

3. Nanoindentation methods

3.1. Classical analysis

To describe the relationship between the response of the indentation tip and the mechanical properties of the materials, the