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Micro-mechanical properties and multi-scaled pore structure of graphene oxide cement paste: Synergistic application of nanoindentation, X-ray computed tomography, and SEM-EDS analysis

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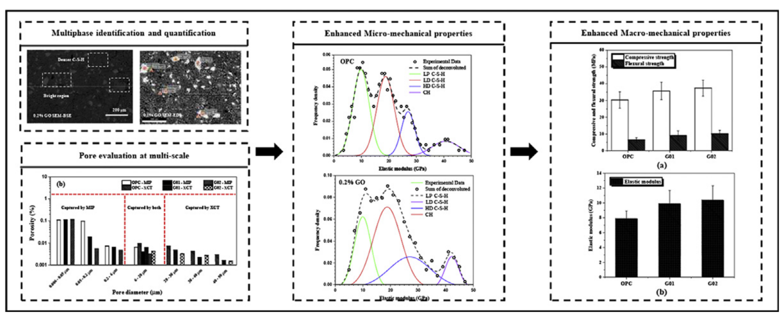
HIGHLIGHTS

- Advanced micro-scale measurements were combined to predict GO cement properties.
- GO enhances both the macro- and micro-mechanical properties of cement paste.
- The use of GO optimizes microstructure and refines pore structure.
- Combination of MIP and XCT facilitates a complete characterization of pore structure.
- Combined tools at multi-scale provide efficient approaches to optimize material design.

GRAPHICAL ABSTRACT



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ABSTRACT

In this study, the microstructure and micromechanical properties of graphene oxide (GO) cement-based materials are investigated. Nanoindentation with statistical deconvolution was used to determine the elastic properties of constituent phases. Besides, combined with scanning electron microscopy (SEM) equipped with energy-dispersive spectrometer (EDS) and backscattered electrons (BSE), each phase of hardened cement paste was identified and quantified as well. X-ray computed tomography (XCT) microscope was applied to determine the morphology of micrometer-size pores, while mercury intrusion porosimetry (MIP) was used to capture the volume fraction of smaller pores (nano-scale). The combined use of XCT and MIP offered an effective approach for further understanding the effect of GO on pore structure at multi-scale. The synergistic analysis of microstructure and micromechanical properties at multi-scale provides valuable information towards the material design of cement-based materials with nanomaterials.

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1. Introduction

Cement-based materials are considered as a heterogeneous and porous composite with multiphase, which containing amorphous crystals, crystalline water and pores from nanoscale to micron size [1]. The structural properties of each size are derived from the structural characteristics of the smaller size, and thus, the damages normally exist and occur in a wide range of scales (from nanoscale to micrometer to millimeter scale). Different types of materials with micron size, such as fly ashes and silica fume, have been widely used in modifying the macro properties of cement-based

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materials [2–5]. Recently, nanotechnology has also been developed in modifying cement-based materials [6–9]. Generally, the applications of nanotechnology in cement-based materials consist of two aspects, one is modifying the properties of cement-based materials from nanoscale by the combination of nanomaterials, and another is using nano-scaled test for further understanding the micro-properties of multiphase in cement matrix.

Graphene oxide (GO) has been reported as a unique nanomaterial with sheets-like structure for effectively improving the macro-properties of cement-based materials [10–13]. Specifically, the toughness of the cement-based materials can be obviously improved by using a small amount of GO sheets, while fragility of cement-based materials is usually contributed to its life cycle reduction and long-term cost increase. Normally, the sheet-like structure of graphene can create a lot more contact area with the host matrix, and furthermore the oxygen functional groups of GO make it exhibit a good interfacial attachment [14,15]. Thus, a few studies have been focusing on the investigation of the interfacial attachment between GO and cement matrix by Alkhateb et al [16] and Abrishami et al [17]. These results indicated that GO is well attached with C—S—H, and its bonding enhancement is one of the major reasons for the toughness reinforcement.

Besides, the oxygen functional groups probably play an important role of the bonding enhancement because the oxygen functional groups can be relatively easy to react in high-alkaline environment with the hydrated products to form the covalent bonds [18,19]. Lin et al [20] considered oxygen functional groups of GO surface can form the adsorbed water storage and transport channel to transport the water molecule, promoting the hydrated reaction of cement-based materials. Wang et al [21] showed that the carboxyl functional groups of GO can react with Portlandite (CH) to generate three-dimensional network structure. Ghazizadeh [22] investigated the effect of GO on the hydration of alite, and found that GO only accelerates the hydration of alite marginally, and can easily aggregate in alite paste. In fact, due to heterogeneity of cement-based materials and various oxygen functional groups, the chemical mechanism in GO cement-based composites still remains unclear.

The micro-mechanical properties of each phase in hardened cement can be obtained with the aid of nanoindentation technique. This technique has been used in characterizing homogeneous materials in the past two decades. By the combination of statistical deconvolution tools, nanoindentation has been increasedly adopted to characterize the heterogeneous cementitious materials with multiphase in the past decade [23–25]. Indeed, statistical nanoindentation technique (SNT) has been proved an effective protocol to calculate the volume fraction of each phase in hardened cement [26–32]. For instance, based on the data of indents, several studies [25,33–35] obtained the relative contribution of low-density and high-density calcium-silicate-hydrates (LD C—S—H and HD C—S—H), Portlandite (CH), and anhydrous clinkers of different systems. Furthermore, synergistic analysis can be conducted for the understanding of each phase hydrated products by the combination of scanning electron microscope (SEM).

Pore structure of the cement-based materials is also important in dictating the macro properties of the heterogeneous material [36,37]. Micro X-ray computed tomography microscope (XCT) techniques is an advanced technique, and can be used for the analysis of pore networks in cementing materials matrix [38]. The principle of XCT is to deal with a large number of 2D images, and overlying 2D images to build up a 3D model. Thus, XCT is considered highly suitable in dictating the transport properties [39–43]. However, due to the technological limitations, the limited resolution of XCT cannot meet the requirement of evaluating the nano-scaled pores. Evaluation of small pores for the cement-based materials is also usually conducted by the mercury intrusion porosimetry (MIP) and nitrogen absorption technique. It is pointed out that air voids, present in pastes unless they are vacuum-mixed, are intruded after the threshold pressure is reached, and are not generally recognized as such in the MIP plots [44]. Hence, the combination of XCT and MIP can prove an effective protocol for the comprehensive evaluation of pore structure of cement-based materials [25].

Based on the studies mentioned above, several studies have been conducted in developing the GO effect in macro properties. However, few studies have been focusing on the GO modified effect in micromechanical properties and porosity structure from multi-scale range at present. Therefore, this study reports a comprehensive investigation of the GO modified effect in micro-mechanical properties of each hydrated phase by coupled using SNT and SEM under backscattered images (BSE) mode. Phase quantification of the heterogeneous microstructure was conducted using the backscattered images mode of SEM. The solid phase volume fractions of the GO cement-based composite were validated by using SNT as well. Energy dispersive spectroscopy (EDS) and the indents in-situ in an SEM under BSE mode were in further carried out for the characterization of each phase of the GO cement-based composite. Besides, the pore structure of the GO cement-based composite was also evaluated combined with XCT and MIP. Three dimensions images for the pore structure of the GO cement-based composite were built up for visual evaluation of the micro pore distribution. In addition, the nano-scaled pore distribution was obtained by MIP. The macro mechanical experiments of GO cement-based composite were also carried out, and the relationship between the macro mechanical properties, phase volume fraction, and the pore structure were analyzed. By the synergistic analysis of microstructure and micromechanical properties for the heterogeneous cementitious materials, this study provides efficient methods to evaluate the macro-mechanical properties, and these efficient methods can also broaden avenues for performance evaluation of similar materials.

2. Materials and methods

2.1. Materials

Ordinary Portland cement PI 42.5R conforming to the requirements of Chinese Standard GB175 [45] is used in this research. The chemical compositions and physical properties are listed in Table 1. The particle diameter range of the cement was scanned by Laser Particle Size Analyzer, as shown in Table 2.

Graphite oxide powder purchased from the Sixth Element Co., Ltd in Changzhou was applied for the preparation of GO nanosheets solution in the experiment. The properties of the Graphite oxide used are given in Table 3. Polycarboxylate-based high-range water-reducing admixture (PC) confirming to the requirements of JG/T223 [46], is used for improving the fluidity of the fresh GO/cement composites. The rate of maximum water-reducing is in the range of 30% to 35%. The characteristics of the chemical admixtures is shown in Table 4.

2.2. Methods

2.2.1. The preparation and characterization of GO

For the GO preparation, the certain amount of graphite oxide was firstly added into deionized water and mixed with a magnetic stirrer for 30 min. Then the aqueous suspension of graphite oxide at the concentration of 5 g/L was sonicated with an Ultrasonic Homogenizer (300 W) for 2 h. The ultra-sonicator was set up in circulation of 2 s working and 4 s intervals in order to avoid suspension overheat. After ultra-sonication, the morphology of GO was verified by employing atomic force microscope (AFM) and transmission electron microscopy (TEM). The chemical bonding of GO was identified by using Fourier transform infrared (FTIR) spectrometer.

2.2.2. Mixing proportions.

For the GO/cement composites fabrication, different PC to GO mass ratio were prepared for ensuring the similar fluidity of all samples. It has been reported that the good dispersion of GO in alkaline cementitious solutions achieved by pre-adding PC [47]. Thus, PC mixed with half portion of the aqueous dispersion was firstly stirred evenly. Then cement was added into the PC/aqueous dispersion,

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