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Electrochemical impedance interpretation for the fracture toughness of carbon nanotube/cement composites

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HIGHLIGHTS

- Fracture behavior of carbon nanotube/cement materials is evaluated by EIS method.
- A novel equivalent circuit model is proposed to explain composite's fracture performance.
- The model parameter R_{ct1} is used to quantitatively assess the fracture toughness of the composite.

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ABSTRACT

Electrochemical impedance spectroscopy (EIS) is adopted as a nondestructive testing method for exploring the fracture feature of the carbon nanotube/cement composite materials. A novel equivalent circuit model is used to explain the change regularity of fracture toughness. Based on the charge transfer on the surface of carbon nanotubes (CNTs), the electrical resistance can be applied to evaluate the conducting efficiency in a certain orientation. The EIS results indicate that more CNTs fibers are perpendicular to the crack opening with increasing content of CNTs, which is corresponding with a higher fracture toughness of CNTs/cement composites. The experimental results also demonstrate the fracture toughness of CNTs/cement composites with different contents (0 wt%, 0.033 wt%, 0.066 wt% and 0.1 wt%) of CNTs could be accurately responded with linear relationship with the EIS results. Furthermore, an advanced equivalent circuit model is proposed to describe the impedance response of CNTs/cement composites. The EIS method therefore can be used as a reliable, convenient and non-destructive method to assess the fracture toughness of CNTs/cement composites.

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1. Introduction

Fiber reinforced cement-based materials are widely utilized as construction and building materials over a few decades, as the fibers can improve the weakness of cement-based materials, such as low ductility, propagation of cracks due to early-age shrinkage. Recently, carbon nanotubes (CNTs), a novel nano-sized fiber, have gained considerable attention in the aspect of fiber reinforced cement-based materials due to its excellent mechanical and physical properties [1–3].

Li et al. [4,5] study the cement-based composites made by adding the CNTs to cement paste and mortar, and found that the flexural strength, compressive strength, freeze-thaw resistance and damping property of cement composites are increased with addition of CNTs. The mechanical and electrical properties of fly ash geopolymeric composites reinforced with CNTs are investigated by Saafi et al. [6]. The experimental results show that the flexural strength, Young's modulus and flexural toughness are increased by 160%, 109% and 275%, respectively. However, a negative effect on mechanical properties is also obtained in Musso et al. [7] research, with an 80% and 60% decrease in compressive strength and rupture modulus, respectively. It has been reported that the effectiveness of fiber in improving mechanical behaviors primarily depends on their dispersion including fiber orientation and arrangement within composites [8]. The orientation or arrangement of CNTs is the primary reason for the contradictory results mentioned above.

Nowadays, CNTs reinforced cement-based materials are typically evaluated for the degree of CNTs dispersion coarsely through comparing their fracture toughness and microstructural characterization. The fracture toughness test, although practical, can only

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qualitatively represent the CNTs dispersion in destined cross-section. In addition, this method belongs to destructive type, in which a large amount of samples need to be destroyed to get the statistic data. What's more, the development of mechanical strength cannot be obtained with the help of this destructive method. Thus, an in-situ nondestructive testing method is needed for evaluating the dispersion of CNTs in cement-based materials.

Among the current testing methods, impedance spectroscopy is a new and powerful method, which helps to characterize the electrical properties of the materials and their interfaces with electronically conducting electrodes. The electrochemical impedance spectroscopy (EIS) method, a non-destructive method, has been applied to study the electrical properties of fiber reinforced cement-based materials [9–13]. Torrents et al. [9,10] firstly confirm the dual-arc impedance response of fiber reinforced cement-based materials based on different failure modes of fiber (pull out and debond) and develop a "fiber-switchable coating model" for the special behaviors of fiber. They find that the ratio between low frequency arc size and the overall resistance could be used as a parameter for characterizing fiber orientation. Wansom and Janjaturaphan [11] detect a relationship between EIS results and flexural strength of fiber/cement composites and demonstrate that the EIS can be applied to evaluate the orientation of short-fiber in plant fiber-reinforced cement-based materials. Wansom et al. [12] also find that EIS method could be used to investigate the CNTs dispersion in nano-composites.

Norman and Robertson [13] investigate the effect of fiber orientation on the toughening of polymers by adding short glass fibers. The results indicate that fibers perpendicular to the flexural plane pull out completely and provide the greatest toughening reinforcement, and fibers parallel to the flexural plane provide the less toughening reinforcement. Hence, it is significant to ensure a larger amount of CNTs become perpendicular rather than parallel to the direction of cracks opening. However, there are still less investigations focusing on the relationship between the CNTs orientation and the fracture toughness of CNTs/cement composites based on the EIS method.

In the research, the EIS method is applied to investigate the fracture toughness of CNTs/cement composites. CNTs with different contents (i.e., 0 wt%, 0.033 wt%, 0.066 wt% and 0.1 wt%) are added to cement paste, which are casted as different specimens for the fracture toughness test. The conducting efficiency of CNTs in the certain orientation is characterized by EIS method, in which the orientation perpendicular to the crack opening. A novel equivalent circuit model is proposed to interpret the fracture behaviors of the cement composites.

2. Experiment

Type I 42.5R Portland cement is used in the research. The purified multi-walled CNTs (offered by Chengdu Organic Chemistry Research Institute) is applied in the experiment. The physical properties of multi-walled CNTs and morphology are shown in Table 1 and Fig. 1, respectively. The poly vinyl pyrrolidone (PVP), which is supplied by the Aladdin Reagents (Shanghai) Co., LTD, is employed as a surfactant to disperse multi-walled CNTs.

The aqueous dispersion of multi-walled CNTs is prepared prior to preparing the CNTs/cement composites. The certain amount of surfactant is added into distilled water and mixed with a magnetic stirrer for 15 min, followed by addition of multi-walled CNTs and further mixing for 10 min. The aqueous suspension of multi-walled CNTs is then sonicated with an Ultrasonic Cell Disrupter (JY92-IIN Ultrasonic cell disrupter, 650 W) for 30 min. The ultra-sonicator is set up in circulation of 3 s working and 3 s intervals in order to avoid suspension overheat.

Table 1
Properties of the multi-walled CNTs used in this study.

Type	Diameter (nm)	Length (μm)	Purity	Specific surface area (m^2/g)	Making method
Multi-walled CNTs	10 ~ 20	10 ~ 30	>95%	>180	CVD

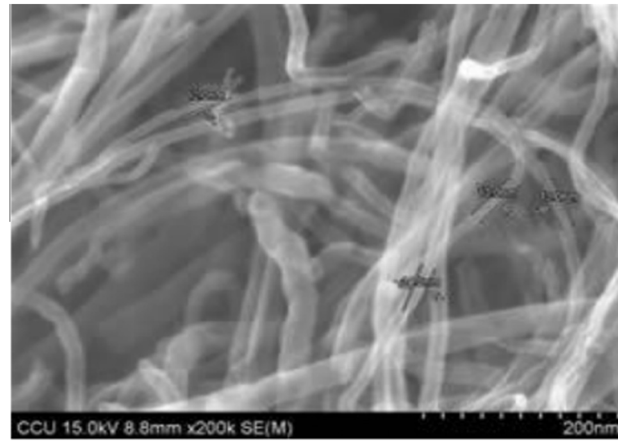

Fig. 1. SEM image of multi-walled CNTs.

Table 2
Mix proportions (g).

Specimen	Cement	Water	Multi-walled CNTs	PVP
C0	100	45	0	0
C1	100	45	0.033	0.132
C2	100	45	0.066	0.264
C3	100	45	0.100	0.400

To prepare a sample, cement is at first added into the aqueous dispersion followed by mixing with a cement mixer for 5 min to get the fresh CNTs/cement composites. Then the fresh CNTs/cement composites are casted for the specimens with two different moulds (30 × 30 × 30 mm and 160 × 40 × 40 mm). The previous specimens are for the electrochemical impedance measurement, while the latter are for the fracture toughness test. Three specimens are prepared for each test. After that, the specimens are demolded after 24 h and placed in the curing chamber (95 ± 5% RH, 20 ± 2 °C). Mix proportions of specimens are shown in Table 2.

The electrical responses of CNTs/cement composites are measured by means of electrical impedance method with PAR Potentiostat/Galvanostat 283. The specimens cured for 3 days and 7 days are carried out for the electrical impedance measurement. Prior to the test, specimens are dried for 18 h at 50 °C. In order to get a good contact between the electrode and the specimen, a wet and thin sponge is placed into the seam between them, as shown in Fig. 2. The electrical impedance measurements are conducted in a frequency range from 0.01 Hz to 1 MHz. The applied electrical field in electrical impedance measurement is perpendicular to the cracks opening (See in Fig. 3).

The fracture toughness of the CNTs/cement composites are toughly tested using a simplified method. This method imitated the draft recommendation proposed by RILEM Committee on Fracture Mechanics of Concrete-Test Methods [14] based on the fictitious model. Small-size specimens that could not fulfill the recommendation were used. It must be noted that the fracture toughness index measured in this way may not be the true values, thus they can only be used to compare each other in this study, rather than with data in other literature. The loading force is restricted in the range of 0–5 KN with the loading rate 0.02 mm/min [15,16]. The specimens 160 × 40 × 40 mm are cut at mid-span with a half depth, as shown in Fig. 3. Three

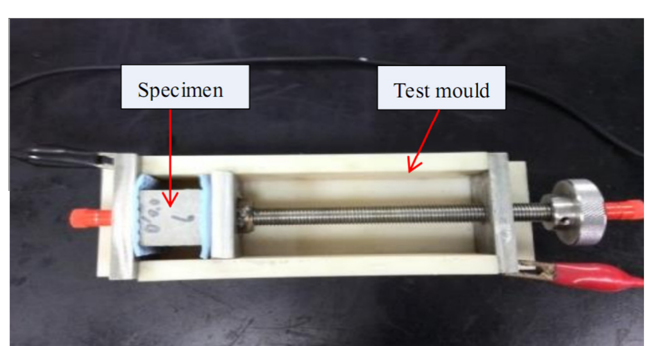

Fig. 2. Specimen and Set-up for electrochemical impedance measurement.

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