

Effect of ultra-fine fly ash on the dielectric behavior of CFSC under stress

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ABSTRACT

Small size particle of ultra-fine fly ash was contributed to the dispersion of carbon fiber in composite in the preparation process. The dispersibility of carbon fiber in composite was associated with self-sensing property of composite. The effect of ultra-fine fly ash on the change in relative dielectric constant of carbon fiber sulphoaluminate cement composite under stress was investigated. The sensitivity, accuracy and reversibility of the change in relative dielectric constant under stress have been improved when 10% content (by mass of cement) of ultra-fine fly ash was added into carbon fiber sulphoaluminate cement composite (CFSC). Besides promoting micro capacitor creation, ultra-fine fly ash could endow CFSC with excellent mechanical property and weaken the ions polarization effect. The combination of above three effects upgraded the dielectric behavior of CFSC under stress. Copyright © 2011 VBRI press.

Keywords: Dielectric; ultra-fine fly ash; sulphoaluminate cement; carbon fiber.



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Introduction

The sensing of stress or strain experienced by a concrete structure is relevant to structural vibration control, traffic monitoring, weighing in motion and hazard mitigation. The self-sensing of stress or strain by a cement-based material is advantageous to sensing by means of embedded or attached sensors (such as strain gages and optical fibers), as it is associated with low cost, high durability, healthy monitoring in real time, large sensing volume and absence of mechanical property degradation. Carbon fiber could endow concrete with self-sensing of stress or strain performance, which has been attained by means of electrical resistivity measurement (i.e., the piezoresistivity effect) [1-12] and voltage measurement (i.e., the direct piezoelectric effect) [13-14]. Besides the above-mentioned measurement means, the relative dielectric constant also could be applied to be the electrical signal of the self-sensing measurement [15-18]. In particular, Sulphoaluminate cement was attractive due to its early high strength, subzero temperature used and low drying shrinkage. Appropriate content of carbon fiber endowed sulphoaluminate cement (CFSC) with stress sensing ability based on its dielectric behavior under stress [18].

Due to the presence of non steady hydration ionic movement, the reversibility of the change in relative dielectric constant under stress of CFSC is not complete. Furthermore, the sensitivity of the change in relative dielectric constant under stress of CFSC needed improvement. The dispersibility of carbon fiber in

composite was associated with the smart property of composite itself. And small size particle of ultra-fine fly ash was contributed to the dispersion of carbon fiber in composite in the preparation process. In present, the effect of ultra-fine fly ash on the dielectric behavior of CFSC was not discussed. The emphasis of this paper is on the effect of ultra-fine fly ash content on the dielectric behavior of CFSC under stress, and the influence mechanism was also investigated.

Experimental

Materials

Short carbon fiber was obtained from Shanghai Carbon Element Corporation. It was isotropic pitch-based and about 5mm length, and its diameter was 7 μm , which was used in the amount of 0.5% (by mass of cement). No aggregate (fine or coarse) was used. The cement used was sulpho- aluminate cement, as obtained from Zibo Cement Corporation, who's compressive and flexure strengths for 3 days were 42.5 and 7.0 MPa respectively. And it's compressive and flexure strength for 28 days was 48 and 7.5 MPa, respectively. The water/cement ratio was 0.35. The ultra-fine fly ash came from Huangtai power plant. Its specific surface area was 652 m^2/kg and its two main chemical components were SiO_2 and Fe_2O_3 , and the content of SiO_2 exceeds one half. The ultra-fine fly ash was used in the amount of 0%, 5%, 10% and 15% (by mass of cement). The methylcellulose was used in the amount of 0.6 % (by mass of cement) in order to disperse short carbon fiber. The defoamer (tributyl phosphate) was used in the amount of 0.2% (by mass of cement) in order to eliminate the bubbles.



Fig. 1. Photograph of neat cement mixer.

Neat cement mixer was used for mixing, whose photograph was shown in **Fig. 1**. Firstly, saturated methylcellulose solution, defoamer, ultra-fine fly ash and short carbon fiber were added into beaker and stirred by glass stick for 2 min. Then the above mixture and water, together with sulphoaluminate cement, were put into the neat cement mixer, and then were mixed and stirred for 5 min. In all cases, after pouring into oiled molds, an external electrical vibrator was used to facilitate compaction and

decrease the amount of air bubbles. The specimens were demoulded after being cured for 1 day under the conditions of 20 \square °C and 95% relative humidity, then curing in water at 20 °C for 28 days. Four types of specimens with different mass of ultra-fine fly ash were prepared, including plain specimen only without ultra-fine fly ash, and were named as 1 $\#$, 2 $\#$, 3 $\#$ and 4 $\#$, respectively.

Testing

Specimens were in the form of rectangular shape. Its two sides were 30 mm, and the other side was 15 mm. A specimen, after polishing on both square faces by 600-grit sandpaper such that the two sides are rendered exactly parallel, was sandwiched by two copper discs (similarly polished) of square face with 30mm side. The copper discs served as electrical contacts. Silver paint was applied between the specimens and each copper disk in order to avoid any air gap at the interface.

Porosity measurements were recorded using mercury intrusion porosimetry (poremaster-60), whose photograph was shown in **Fig. 2**, in the 3.5-200000 nm range. Before measurements, each sample was placed in a special cell and was vacuum pumped to 10 Pa. The vacuum was applied for approximately 1 h to ensure that all the air and residual moisture were removed from the pores. Mercury porosimetry measurements are based on the gradual intrusion of mercury to the pore system of the mixture, during the application of an external pressure. The pore radius is then calculated as function of pressure using the Washburn equation.



Fig. 2. Photograph of mercury intrusion porosimetry.

The impedance (4294A), whose photograph was shown in **Fig. 3**, was measured along the 15mm side of the specimen using the two-probe method at frequencies ranging from 1 kHz to 110 MHz. The resistance and reactance were obtained from the impedance by assuming that they were in series connection. The capacitance was obtained from the reactance. The dielectric constant was

obtained from the capacitance. Six specimens of each type were tested in order to ensure statistical significance to the data in spite of possible nonuniformity in the fiber distribution. To show that the dielectric constant measurement using the method described above was accurate, measurement was made on standard sample. The known dielectric constant of standard sample is 10.3 at 1 kHz. Measurement in this work at 1 kHz gave a value of 10.3 also



Fig. 3. Photograph of the impedance.

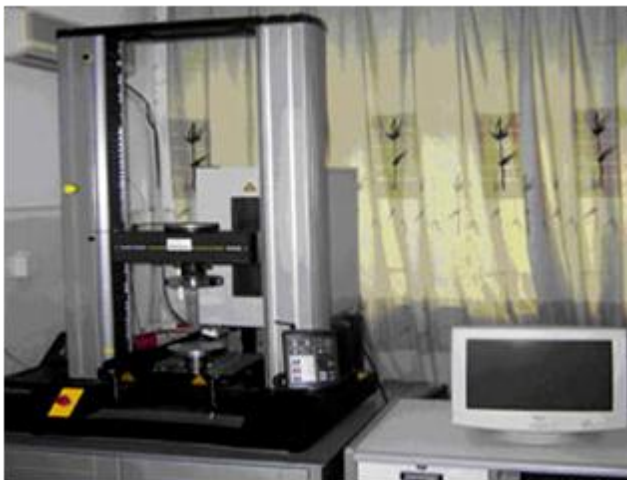


Fig. 4. Photograph of universal testing machine.

For testing the dielectric behavior, during the impedance measurement, compressive stress was applied to the sandwich so that the stress was parallel to the direction of impedance measurement. The stress (repeated loading at increasing stress amplitudes within the elastic regime) was provided by a universal testing machine (INSTRON-5569), whose photograph was shown in Fig. 4. Repeated loading was used in order to investigate the reversibility of the effect. Six specimens of each type were tested.

Results and discussion

Four types of specimens with different ultra-fine fly ash content were prepared in experiment and their dielectric behavior under uniaxial stress was measured. The effects of

ultra-fine fly ash content on the dielectric behavior under stress were illustrated in Fig. 5. For that the compressive strength of the specimen with 15% ultra-fine fly ash was much slower than others, its dielectric behavior under stress was not illustrated in the Fig. 5. As shown in Fig. 5, when ultra-fine fly ash content was smaller than 15%, the fractional change amplitude in the relative dielectric constant of the specimens increased with ultra-fine fly ash content rising. This was related to that ultra-fine fly ash could improve the dispersibility of carbon fiber in sulphoaluminate cement. Firstly, in the preparation process ultra-fine fly ash could weaken viscosity of sulphoaluminate cement paste. Secondly, ultra-fine fly ash has some properties of soft surface, spherical shape as well as low adsorption for water. These two factors would do well to the dispersibility of carbon fiber in sulphoaluminate cement. The dispersibility of carbon fiber was intimately associated with the dielectric behavior of CFSC. Carbon fiber cement composite contained many realized micro capacitors [19] which were consisting of two pieces of carbon fiber and the sulphoaluminate cement between them. The realized micro capacitor was shown from Fig. 6. The better dispersibility of carbon fiber in composite, the number of the micro capacitor was bigger. When carbon fiber cement composite was stressed under force, the electrode distance of the realized micro capacitor would become smaller and the capacitance of the realized micro capacitor [19] would increase under stress. So the good dispersibility of carbon fiber would lead the capacitance for the whole composite to increase much higher. The sensitivity of the dielectric behavior under stress was accord with the change amplitude of the relative dielectric constant under stress. Therefore, the sensitivity of the change in relative dielectric constant under stress has been improved when appropriate ultra-fine fly ash was introduced into CFSC.

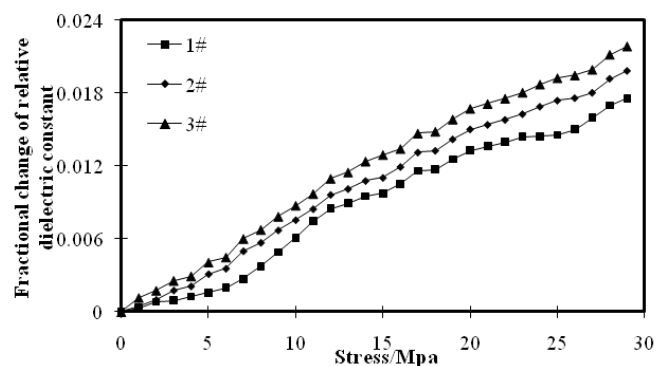


Fig. 5. Effect of ultra-fine fly ash content on the change of relative dielectric constant of CFSC under uniaxial stress.

The Fig. 7 showed the fractional changes in the relative dielectric constant and time of 1[#] and 3[#] specimens, when the specimens were stressed on the repeated compressive stress for 10 cycles and the maximum value of the repeated stress was 10 MPa. As shown from Fig.5 and Fig.7, it was apparent that the linearity of the change in relative dielectric constant under uniaxial effect was improved when appropriate ultra-fine fly ash was mixed into composite. This was related to that ultra-fine fly ash

could weaken the ions polarization effect which would interfere with the accuracy of electrical signal measurement. It is well known that there are many both positive ions (such as K^+ , Na^+ , Ca^{2+}) and negative ions (for example OH^- , SO_4^{2-}) in carbon fiber cement composite. Most of these ions existed in interface area of CFSC. These ions transferred and then came into being ion conductance under external electron field. However positive and negative ions would transfer to opposite directions. This resulted in ions enrichment on the two electrodes. When a new electron field was produced, it was opposite to the external field which was so called the ions polarization effect. The ions polarization effect could interfere with the accuracy of electrical signal measurement. The addition of ultra-fine fly ash resulted in the reduction of water demand of preparation for CFSC, which was unfavourable to the formation of high interface ions concentration in the interface area. Furthermore, the liquid channel through which ions could transfer would be clogged when ultra-fine fly ash was introduced into CSFC for its small crystal size. These above-mentioned factors could weaken the effect of ions polarization effect on the measure electrical signal. It was concluded that ultra-fine fly ash could improve the accuracy of the measure signal of the change in the relative dielectric constant under uniaxial stress.

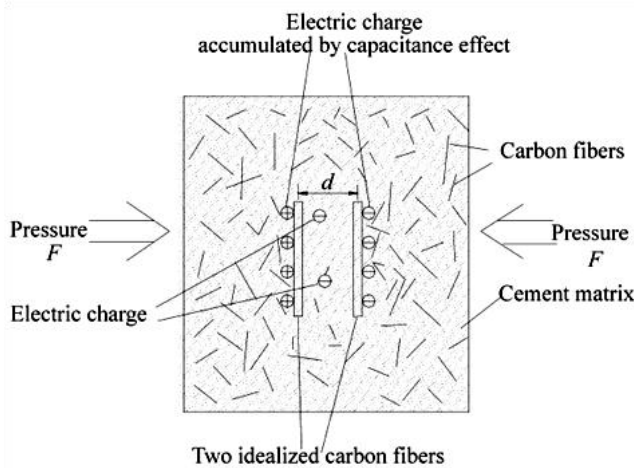


Fig. 6. Realized micro capacitor in CFSC.

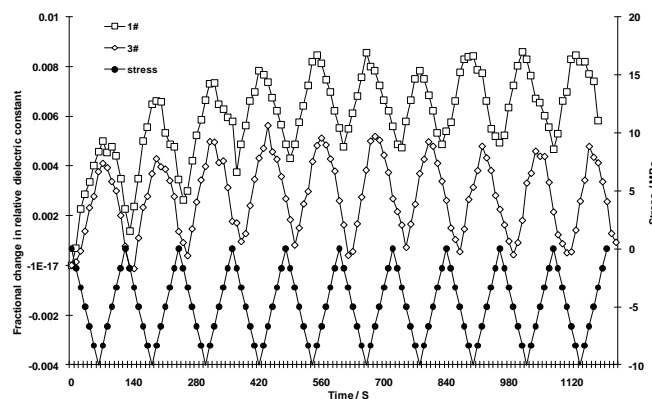


Fig. 7. Fractional change of the relative dielectric constant and time of 1[#] and 3[#] specimens under the repeated compressive stress for 10 cycles.

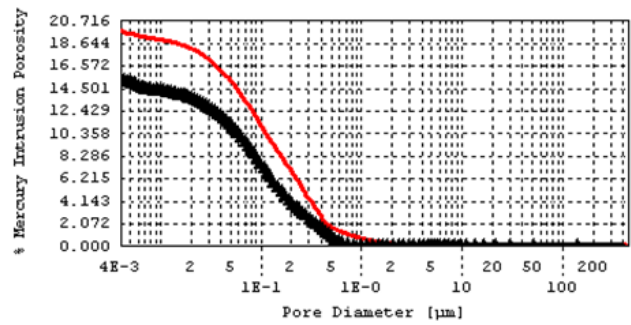


Fig. 8. Pore structure and porosity of 1[#] and 3[#] specimens.

Only under repeated stress the relative dielectric constant showed accordingly regular variations, CFSC should have promising to be prepared into stress sensor for stress sensing. Fig. 7 illustrated the reversibility of the relative dielectric constant under repeated stress for 1[#] and 3[#] specimens. As shown from Fig. 7, the stress decreased back to zero at the end of each compressive stress cycle. The fractional changes of the relative dielectric constant of 1[#] and 3[#] specimens decreased reversibly upon compressive stress in each cycle. The higher the stress amplitude, the greater was the extent of the change of the relative dielectric constant rising. As load cycling progressed, the fractional change of the relative dielectric constant for 1[#] specimen at zero stress increased gradually cycle by cycle in the first five cycles, which indicated that some damage appear under stress and the change in relative dielectric constant under repeated stress became stable in the following cycles. And for 3[#] specimen the fractional change of the relative dielectric constant kept good reversibility all the long time. It was concluded that the reversibility of the relative dielectric constant under repeated stress of 3[#] specimen was better than that of 1[#] specimen.

In order to disperse carbon fiber, the methylcellulose was added into cement paste, which would introduce much air into cement paste and many pores would produce in the specimens. In addition, small air-bubbles were created in mixed process. Ultra-fine fly ash could decrease viscosity of sulphoaluminate cement paste in preparation process. The lower viscosity of cement paste would prevent pores from coming into being in specimen. Furthermore, ultra-fine fly ash could be filled in the hole and gap between sulphoaluminate cement hydration products. Fig. 8 was the pore structure and porosity of 1[#] and 3[#] specimen, showing that the porosity of CFSC with 10% ultra-fine fly ash was much smaller than that of CFSC without ultra-fine fly ash. This intensified that ultra-fine fly ash improved the impaction of CFSC. Hence, ultra-fine fly ash could endow CFSC with excellent mechanical property. On the other hand, as already noted, the addition of ultra-fine fly ash resulted in the good dispersibility of carbon fiber in composite, which was favorable to the formation of the micro capacitor. Under stress the capacitance of the micro capacitor would increase. In this case the micro capacitor would catch nearby ions, which resulted in decreasing the ions movement under stress. As has been said, excellent mechanical property and decreasing the ions movement would contribute to the reversibility of the change in relative dielectric constant under repeated stress. It was concluded that appropriate content of ultra-fine fly ash

could improve the reversibility of the dielectric behavior of CFSC under repeated stress.

Conclusion

Sulphoaluminate cement was attractive due to its early high strength, subzero temperature used and low drying shrinkage. Appropriate content of carbon fiber endowed sulphoaluminate cement with stress sensing ability based on its dielectric behavior under stress. Whereas the reversibility and sensitivity of the change in relative dielectric constant under stress of CFSC needed improvement. Ultra-fine fly ash could promote micro capacitor creation, weaken the ions polarization effect and endow CFSC with excellent mechanical property. The sensitivity, accuracy and reversibility of the change in relative dielectric constant under stress have been improved when 10% content (by mass of cement) of ultra-fine fly ash was added into CFSC.

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