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Effect of preparation processes on the piezoresistivity effect of CFSC

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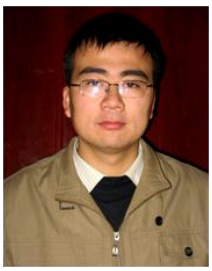
ABSTRACT

The preparation process of carbon fiber sulphoaluminate cement composite (CFSC) was intimately associated with its piezoresistivity effect. In this paper the piezoresistivity effects of CFSC prepared by different preparation processes were investigated. The experimental results indicated there was no significance difference of the variation amplitude for all specimens. Meanwhile, the good reversibility order of the piezoresistivity effect for all specimens was the specimen prepared by pressing, the specimen prepared by extrusion and the specimen prepared by casting. This phenomenon was related to the interfacial layer structure. Therefore, pressing and extrusion technology could improve the reversibility of the piezoresistivity effect of CFSC. Copyright © 2011 VBRI press.

Keywords: Carbon fiber; sulphoaluminate cement; pore; piezoresistivity effect.



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Introduction

Vibration reduction is needed for most structures, due to the resulting improvement in performance, control and safety. Associated with vibration reduction is the sensing of vibration, which relates to the sensing of load. Moreover, the monitoring of the load history is useful for determining the cause of damage of a structure. An emerging and attractive method of strain sensing involves using the structural material itself as the sensor [**1-2**]. This means that there is no attached or embedded sensor. This method is known as self-sensing and is attractive because of its low cost, high durability, good compatibility and no degradation of mechanical property. The ability of structural materials to sense their own strain has been reported in short carbon fiber cement-matrix composites based on their piezoresistivity effect [**3-14**]. The self-sensing ability in these composites is based on piezoresistivity, which is the phenomenon in which the electrical resistivity of the material variations reversibly with strain. In contrast, the metal film strain gages are not based on piezoresistivity, since the resistivity of the metal does not change with strain.

Appropriate content of carbon fiber endowed CFSC with stress sensing ability based on its piezoresistivity effect [**15-18**].Sulphoaluminate cement endowed CFSC with good frost-resistance and low drying shrinkage. Due to good frost-resistance, CFSC has advantage to be used in the building in cold area, for example the Qinghai-Tibet Plateau, where the temperature was almost under zero in the whole year. Low drying shrinkage performance of CFSC would not produce extra stress on itself which would

interfere with the accuracy of stress self-sensing. However, the peizoresistivity effect of CFSC was required to be refined in order to satisfy real stress sensing. In particular, the reversibility of the piezoresistivity effect should be improved, which was related to the interfacial structure of the composite. In this paper, CFSC was prepared by three kinds of preparation process. The interfacial structure and its smart property of composite would be different if the preparation process was different. In order to find the method to improve the reversibility of the piezoresistivity effect, the relationship of smart property of composite and its interfacial structure would be discussed in this paper.

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 7μm, 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, whose compressive and flexure strengths for 3 days were 42.5 and 7.0 MPa, respectively. And its compressive and flexure strength for 28 days were 48 and 7.5 MPa, respectively. The water/cement ratio was 0.42. 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 0.3% (by mass of cement) in order to eliminate the bubbles. The plasticizer (DOP) was used in the amount of 0.5% (by mass of cement) in order to add the plasticity of specimen in extrusion process.

Fig. 1. Photograph of neat cement mixer.

Fig. 2. Photograph of vacuum pug mill.

Three preparation processes were list as following.

1. Casting process: Neat cement mixer was used for mixing, whose photograph was shown in **Fig. 1**. Firstly, saturated methylcellulose solution, defoamer 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. 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 and cut into the form of rectangular shape of size 30 × 30 × 15mm^3 after being cured for 1 day under the conditions of 20 ^{o}C and 95% relative humidity.
2. Extrusion process: Firstly, saturated methylcellulose solution, defoamer and short carbon fiber were added into beaker and stirred by glass stick for 2 min. Then the above mixture, water, plasticizer, together with sulphoaluminate cement, was put into the neat cement mixer, and then were mixed and stirred for 5 min. Finally, the mixture was put into vacuum pug mill (whose photograph was shown in **Fig. 2**) to extrude and cut into the form of rectangular shape of size 30 × 30 × 15mm^3.
3. Pressing process: Short carbon fiber, ethanol (not only help to disperse carbon fiber in cement, but also could prevent cement from rapid hydrating) and sulphoaluminate cement were put into the beater, mixed and stirred by glass stick for 2 min. Then the mixture was dried in oven at 20 ^{o}C for 5h to get rid of ethanol. After that appropriate water was mixed into the dried mixture. In this case the ration of water/cement was only 0.2, for that cement added much water would become paste and not to be pressed. Finally, the mixture was pressed with stress of 5 MPa in compacting tool set and cut into the form of rectangular shape of size 30 × 30 × 15mm^3.

Fig. 3. Photograph of the impedance.

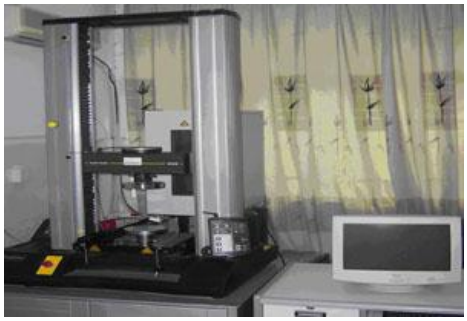

Fig. 4. Photograph of universal testing machine.

In all cases, all kinds of specimens were cured in water at 20 °C for 28 days. Three types of specimens prepared by different preparation process and were named as1#, 2# and 3#, respectively. In order to eliminate the influence of humidity content of the cement paste on the measurement result, all specimens were dried for 24 h at an elevated temperature of 105 °C under the vacuum condition of 0.05 MPa, which were offered by vacuum drying chamber.

Testing

Specimens were in the form of rectangular shape. Its two sides were 30mm, 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 30 mm 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.

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 was obtained from the impedance by assuming that they were in series connection. 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 resistance measurement using the method described above was accurate, measurement was made on standard specimen. The known resistance of standard specimen is 5.6 Ω at 1 kHz. Measurement in this work at 1 kHz gave a value of 5.6 Ω also.

For testing the piezoresistivity 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. The microstructures of the specimens were analyzed by scanning electron microscope (S-2500).

Results and discussion

The Electrical conduction in CFSC involved electrons and ions. In the water saturated state, ionic conduction dominated; whereas in the dry state, electronic conduction was more significant than ionic conduction. Carbon fiber is an electronic conductor and the charger carriers would be electrons and holes. While in the dry state, percolation theory and tunneling effect theory are usually employed to interpret the conduction mechanism of CFSC. An idealized model of percolation was that adjacent fibers physically contacted and formed a geometrically connected phase when the fiber content was near or above the percolation threshold. However, the tunneling effect also contributed to the conduction for the case when two adjacent fibers were not physically in contact, even when the fiber content was below the percolation threshold. In general, a low volume fraction of fibers (usually near the percolation threshold) is preferred in practice not only since lower cost, good workability and higher compressive strength (low air void content) can be obtained, but also a decrease of fiber addition close to the percolation threshold will increase the sensitivity of CFSC as a stress sensor (usually based on the piezoresistivity effect). Good sensitivity of the piezoresistivity effect means the obvious variation amplitude of the resistivity with stress changing. Except for good sensitivity, good reversibility was another desired performance for the piezoresistivity effect.

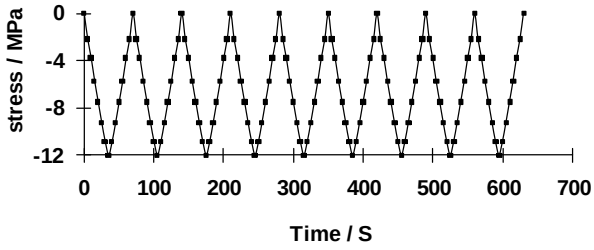

Fig. 5. Applied stress and time of all specimens during repeated compressive stress.

Fig. 5 showed the applied stress and time during repeated compressive stress. The variations in resistivity and time of all the specimens during repeated compressive stress was illustrated in **Fig. 6**. In the experiment the amplitude of repeated compressive stress was 12 MPa. Under above repeated compressive stress, the deformation of CFSC was elastic deformation for that the failure strength of CFSC was about 40 MPa. As shown from **Fig. 6**, the variations in resistivity of all the specimens were corresponding to the cycle stress variation, and the greater the compressive stress amplitude, the more resistivity increases. This showed that all the specimens prepared by different preparation process had obvious piezoresistivity effects. Excellent piezoresistivity effect of all specimens was contributed to the appropriate content of carbon fiber content in CFSC, which was close to the percolation threshold. However, from **Fig. 6** it was also seen that the variation amplitude order of the piezoresistivity effect for all specimens during cycle stress was 1# specimen, 2# specimen and 3# specimen. Meanwhile, some irreversible variation in resistivity of 1# specimen appeared, but these variations were not obvious in 2# and 3# specimens. The reversibility order of the piezoresistivity effect for all specimens during cycle stress was 3# specimen, 2# specimen and 1# specimen.

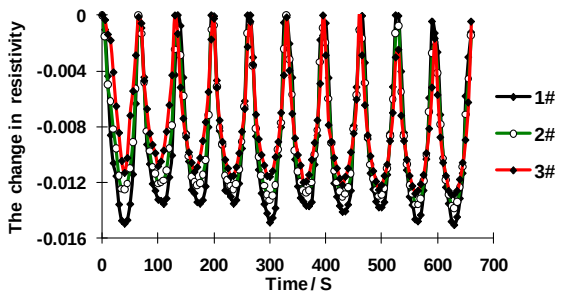

Fig. 6. Variation in resistivity and time of all specimens during repeated compressive stress.

The variation amplitude of the piezoresistivity effect of CFSC was related to the concentration variation of the effective charge carrier in the tunneling effect during repeated compressive stress, which was happened between two pieces of adjacent realized carbon fiber in CFSC composites. The concentration of the effective charge carrier (n) [**19**] could be shown as formula (1).

$$n = n_0\left(\frac{4E}{U_0}\right)^{N_0} e^{-\frac{2a}{h}N_0\sqrt{2m(U_0-E)}} \qquad \text{---------- (1)}$$

where, n_0, N_0, U_0, a, h and E are the total number of charge carriers, number of barriers connected in series, height of the barrier, width of the barrier, reduced Plank's constant and the energy of the charge carrier, respectively.

Except for a, n_0, N_0, U_0, h and E is constant under stress in formula (1). The variation of a under stress was decided by the applied stress and the elasticity modulus of specimen. The elasticity modulus of specimen was intimately associated with porosity of CFSC. The bigger the porosity content, the smaller was the elasticity modulus of the specimen. Under the same applied repeated compressive stress, the smaller elasticity modulus means that the bigger variation of a parameter. This resulted in the bigger variation of effective charge carrier in the tunneling effect during repeated compressive stress. It was concluded that the bigger the porosity content, the bigger was the variation amplitude of the piezoresistivity effect of the specimen. The porosity and pore structure of all the specimens were illustrated in **Fig. 7**. As shown from **Fig. 7**, the porosity content order of all specimens was $3^{\#}$ specimen, $2^{\#}$ specimen and $1^{\#}$ specimen. So the variation amplitude order of the piezoresistivity effect for all specimens during cycle stress was $1^{\#}$ specimen, $2^{\#}$ specimen and $3^{\#}$ specimen. But in one words, there was no significance difference the variation amplitude for all specimen.

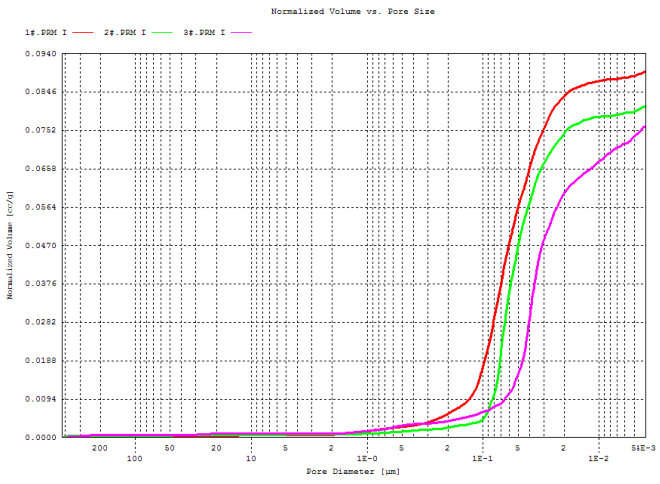

Fig. 7. Porosity and pore structure of all the specimens.

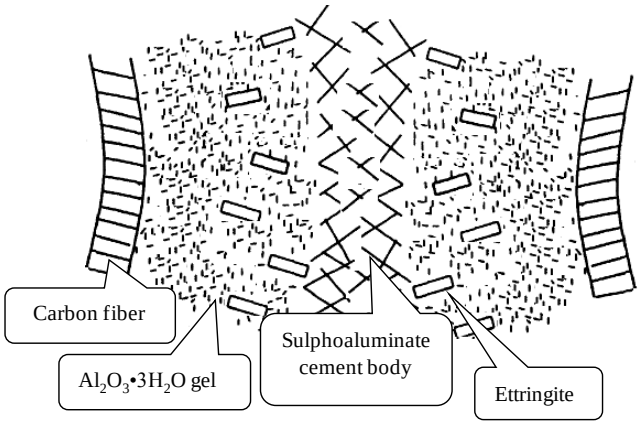

Fig. 8. interfacial structure pattern between two pieces of carbon fiber.

The reversibility of the piezoresistivity effect for the specimen was related to the interfacial structure. **Fig. 8** was the interfacial structure pattern between two pieces of carbon fiber. As shown from **Fig. 8**, the interface layer lain between carbon fiber and sulphoaluminate cement body. Its thickness was about in the range from 10 μm to 50 μm. In the interfacial area, Al_2O_3•$3H_2O$ gel was close to the carbon fiber, and ettringite crystal was close to the Al_2O_3•$3H_2O$ gel. Al_2O_3•$3H_2O$ gel had a better bonding state with carbon fiber. But the area of ettringite crystal concentration has a less dense interfacial structure, where the porosity was high and was considered the weakest link in the interfacial lay. The reason of the interfacial layer producing was that a water film layer appeared in the preparation process, and ettringite crystal easily concentrated in this area.

The interfacial structure was related to the preparation process. Because the casting specimen was prepared by no pre-stress, its interfacial layer thickness would be the biggest, and the porosity of the interfacial layer would be the highest and its interfacial compactness was the loosest among all specimens prepared by different preparation process. Meanwhile, the sulphoaluminate cement body between two pieces of carbon fiber would also be looser in specimen prepared by casting than that of the specimens prepared by extrusion and pressing. On the contrary, the interfacial layer and the sulphoaluminate cement body between two pieces of carbon fiber of the specimen prepared by pressing was the densest among all the specimens.

Tab. 1. Interfacial porosity of the specimens prepared by different preparation process.

Specimen type	Cement matrix density	Carbon fiber density	Composite density	Interfacial porosity
$1^{\#}$	/cm^3	/cm^3	/cm^3	8.0%
$2^{\#}$	/cm^3	/cm^3	/cm^3	5.0%
$3^{\#}$	/cm^3	/cm^3	/cm^3	4.0%

As shown from **Fig. 6**, some irreversible variations in piezoresistivity effect of $1^{\#}$ specimen were illustrated apparently, but these variations were not obvious in $2^{\#}$ and $3^{\#}$ specimens. The irreversible variation was attributed primarily to minor damage in the interface area. The interface area between carbon fiber and sulphoaluminate

cement was the weakness area in the composite. The damage would produce almost in the interface area under cycle stress. The bigger the interfacail porosity value, the easier the interfacial layer was destroyed under the repeated stress. Extrusion and pressing process would improve the interfacial compactness, which would reduce the interfacial damage of CFSC. Interfacial porosity of the specimens prepared by different preparation process was illustrated in **Tab. 1**. The density difference between composite and its matrix originated approximately in the interfacial porosity and introduction of carbon fiber which substituted for the same volume sulphoaluminate cement. In term of the densities of matrix, composite and carbon fiber, the interfacial porosity was calculated by measuring the density of the all sorts of composite and their corresponding matrix. Because the content of carbon fiber was far smaller than the sulphoaluminate cement body, the density difference created by introduction of carbon fiber could be negligible. As shown from Tab.1, the porosity of specimen prepared by extrusion was smaller than that of specimen prepared by casting, and the porosity of specimen prepared by pressing was smaller than that of specimen prepared by pressing. So the reversibility order of the piezoresistivity effect for all specimens during cycle stress was $3^{\#}$ specimen, $2^{\#}$ specimen and $1^{\#}$ specimen.

Conclusion

Sulphoaluminate cement was attractive due to its early high strength, subzero temperature used and low drying shrinkage. Appropriate content of carbon fiber endowed cement with stress sensing ability based on its piezoresistivity effect. Whereas the reversibility of the variation in piezo- resistivity effect of carbon fiber sulphoaluminate cement composite has to be improved. It was found that extrusion and pressing technology could improve the reversibility of the variation in piezoresistivity effect, although the variation amplitude of the piezoresistivity effect decreased a little. This was related to the compactness of the interfacial layer structure of the composite. Extrusion and pressing technology have advantage to be used into the preparation of carbon fiber sulphoaluminate cement composite. In this condition, carbon fiber sulphoaluminate cement composite only could be fabricated into small stress sensing sensor. The big volume stress sensing sensor could not be prepared by pressing and extrusion technology.

Acknowledgement

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