



Technical Note

Freezing–thawing effects on electromagnetic wave reflectivity of carbon fiber cement based composites

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HIGHLIGHTS

- Specimens of the composites with different carbon fiber contents were prepared.
- The electromagnetic wave reflectivity of the composites was investigated and analyzed.
- The microstructure and the pore size distribution changes were analyzed.
- Technical measurements enhancing wave-absorbing ability were recommended.

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ABSTRACT

Electromagnetic pollution has increasingly attracted attention with the development of electronic technology. Carbon fiber cement based composites are widely used as electromagnetic wave absorbing material. However, freezing–thawing cycles can affect their wave absorbing function. In this paper, specimens of the composites with different carbon fiber (CF) contents were prepared. The reflectivity variation of the composites against the wave was investigated before and after 50 freezing–thawing cycles by Naval Research Laboratory (NRL) testing system in 2.0–18.0 GHz frequency bands. The microstructure and the pore size distribution were analyzed by Scanning Electron Microscopy (SEM) and a pore distribution analyzer. Results show that the reflectivity varies slightly before and after freezing–thawing without any CF. In the lower frequency bands, the reflectivity of the composites with different CF contents increases obviously after freezing–thawing cycles. In the higher frequencies, it decreases first and increases then with the minimum value of -12.5 dB and 2.5 GHz frequency band. When the CF content is higher than 0.2%, freezing–thawing cycles can decrease the wave absorbing property of the composites. The porosity of the composites decreases with the increase of the CF contents. It rises when the CF content is 0.8 wt.%. Lower porosity is beneficial to improve the electromagnetic wave absorbing property for the composites.

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1. Introduction

With the development of electronic information technology, electromagnetic interference and microwave pollution have increasingly attracted attention. Cement based composites are widely used as structural materials. Their microwave absorbing properties and electrical conductivity are very weak. However, carbon fiber cement based composites are good smart structural materials with good durability, reliability, and high sensitivity. They can cover the scope of elastic and plastic strains and obtain wide application in the microwave shielding works [1,2]. Research-

ers have studied the reflectivity of the composites with regards to the carbon fiber treatment process [3], the structure of the composites [4], the microwave shielding test methods [5,6], etc., to evaluate the anti-wave interference.

However, cement based composites have some absorbent property. When they are in the environment of moisture for a long term, there must be some water inside. Ice can be formed in negative temperatures, leading to volume expansion. When the stress caused by this swelling is over the strength of the composites, cracks occur and water absorption increases. Such multiple freezing–thawing cycles result in the damage of the materials in the end. Carbon fiber cement based composites are often in open air environment whether they are used either as structural engineering materials [7], temperature and stress sensors [8,9] or as structural reinforcement materials. After freezing–thawing cycles in

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winter, the internal composition and structure were affected. The mechanical properties [10,11], durability [12,13] and electrical properties [14] deteriorate seriously. The electromagnetic absorption properties are also changed.

Currently, a melting agent is usually adopted to remove snow on the cement concrete pavement, which affects the chemical composition changes inside concrete [15]. Therefore, different countries have launched experimental researches on melting and removing snow on the road surface by use of carbon fiber cement based composites. The composites experience the process of power-off and power-on repeatedly during freezing and thawing. After frequent freezing–thawing cycles, the conductivity changes greatly [16], which is closely associated with reflectivity. Reflectivity is an important parameter to evaluate the wave absorbing property of a material. The lower the reflectivity, the stronger the microwave absorption. When reflectivity is less than -10 dB, the material mainly absorbs electromagnetic waves [1,3].

In the present work, the electromagnetic reflectivity variation of carbon fiber cement based composites against electromagnetic waves after freezing–thawing cycles was investigated. The influences of CF contents and porosity over the reflectivity were analyzed. The microstructure of the composites was studied by SEM. The pore size distribution changes were analyzed by a pore distribution analyzer. The primary scope of this study is to interpret how CF contents in the composite before and after freezing–thawing cycles affect the electromagnetic wave absorbing property to put forward theoretical basis and technical measurement for improving the quality and durability of carbon fiber cement based composites in electromagnetic shielding engineering.

2. Raw materials and test program

2.1. Raw materials

Cement used was 42.5R ordinary Portland cement. The main physical and chemical properties are listed in Table 1. Locally available natural sand with particles smaller than 4.75 mm, fineness modulus of 2.39, and specific gravity 2.60 g/cm^3 , was used as fine aggregate. Chopped short carbon fibers were used. The technical parameters of which are shown in Table 2. Sodium carboxymethyl cellulose was used as a dispersing agent. The mass fraction of the aqueous solution was 0.1%. Tap water was used as mixing water.

2.2. Sample preparation and test

2.2.1. Preparation of the composites

Carbon fiber contents (by cement mass) were 0.2%, 0.4%, 0.6% and 0.8%. The ratio of water:cement:fine aggregate was 1:2:6. Flow chart of molding method of the composites was shown in Fig. 1. The weighed dispersion agent and water were added in a beaker. A glass rod was used for stirring for 60 s. Carbon fibers were then added and the dispersion agent was continuously stirred and ultrasonic vibration was conducted for 120 s for uniform dispersion. A JJ-5 cement mortar mixer was used for mixing. Cement and sand were added and mixed 30 s and 120 s, respectively. The stirred mixture was poured into a $180 \text{ mm} \times 180 \text{ mm} \times 10 \text{ mm}$ steel model and vibrated for 30 s in a vibration platform. The surface of the model was leveled in a zigzag way by a trowel. The model was placed in a standard curing box, the temperature inside which was $20 \pm 2 \text{ }^\circ\text{C}$ and the relative humidity was over 90%. The model was removed after 24 h. The specimen was put into $20 \text{ }^\circ\text{C}$ water for curing for 28 days for subsequent use.

2.2.2. Freezing–thawing cycle tests

In accordance with *Standard for Test Methods of Long-term Performance and Durability of Ordinary Concrete (GB/T 50082-2009)*, 50 freezing–thawing cycle tests were carried out with different CF contents and different water–cement ratios. The freezing and thawing temperatures were $-20 \text{ }^\circ\text{C}$ and $20 \text{ }^\circ\text{C}$, respectively. Each freezing–thawing cycle needed 8 h.

Table 1
Properties of ordinary Portland cement.

Setting time/min		Strength at 3 curing days/MPa		Strength at 28 curing days/MPa		Main chemical components/%			
Initial setting	Final setting	Flexural strength	Compressive strength	Flexural strength	Compressive strength	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃
131	302	4.9	28.4	6.8	50.4	60.17	20.85	6.29	5.97

Table 2
Properties of carbon fiber.

Items	Parameters
Carbon content/%	95.5
Stretching modulus/GPa	228
Resistivity/ $(\Omega \text{ cm})$	1.6×10^{-3}
Length/mm	3–5

2.2.3. Electromagnetic wave reflectivity test

Each group consisted of three specimens for the measurement of reflectivity. The specimen, together with another piece, was placed in a drying oven with the temperature of $60 \text{ }^\circ\text{C}$ till constant weight. The Naval Research Laboratory (NRL) testing system was employed for reflectivity test. The system was equipped with a network analyzer. The frequency ranged between 2.0 and 18.0 GHz. The signal was sent off from the network analyzer by one horn antenna, projected onto the sample, and was reflected. The other horn antenna received the reflected signal through the network analyzer for analysis. The test method in details can be referred in [1,17,18].

2.2.4. Microstructures and pore distribution tests

Scanning Electron Microscopy (SEM) was applied for the morphology analysis of the composites. The test conditions were the 20 kV low vacuum mode with a resolution of 3.5 nm. In addition, the porosity of the composites after freezing–thawing test was measured by a pore distribution analyzer. The pore size ranges between 3.5 Å and 5000 Å.

3. Results and discussion

3.1. Reflectivity of the composites with different CF contents

Fig. 2 shows that in the 2.0–4.0 GHz frequency ranges, the minimum reflectivity of -2.2 dB appears at 2.5 GHz before freezing–thawing test when there are no carbon fibers added. After freezing–thawing, the minimum reflectivity of -3.3 dB takes place at 3.8 GHz. The two reflectivity curves coincide. In either case, electromagnetic waves are reflected. When carbon fibers are incorporated, the reflectivity decreases significantly after freezing–thawing cycles. Particularly, when the fiber content is 0.2%, an absorbing peak with the reflectivity of -11.0 dB occurs at 3.4 GHz. At this movement, the composites mainly absorb electromagnetic waves, though the frequency width is only 0.2 GHz.

In the 4.0–8.0 GHz frequency bands, there are no obvious changes in reflectivity before and after freezing–thawing when there are not carbon fibers doped. The reflectivity curves in the two cases coincide. When carbon fibers are incorporated, the reflectivity decreases before freezing–thawing cycles. The minimum reflectivity of -9.4 dB appears at 8.0 GHz with the CF content of 0.4%.

In the 8.0–12.0 GHz frequency bands, the reflectivity of the composites before freezing–thawing cycles is lower than that after freezing–thawing. The minimum reflectivity of -11.5 dB occurs at 8.6 GHz when the carbon fiber content is 0.4%. The effective frequency band width is 2.0 GHz and the composites absorb electromagnetic waves.

In the 12.0–18.0 GHz frequency bands, the maximum reflectivity of -5.4 dB appears at 17.6 GHz when the CF content is 0.2%. When the CF content is 0.4%, the minimum reflectivity of -12.5 dB takes place at 15.0 GHz. The effective frequency band width is 2.5 GHz and the composites show strong absorption of electromagnetic waves. When the CF content reaches 0.8%, the

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