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Enhanced electromagnetic interference shielding of carbon fiber/cement composites by adding ferroferric oxide nanoparticles

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HIGHLIGHTS

- The introduction of nano-Fe_3O_4 into CF/cement composites could significantly enhance the EMI SE.
- The incorporation of 5 wt% nano-Fe_3O_4 along with 0.4 wt.-% CF in the cement led to a SE of 29.8 dB.
- The excellent EMI SE could be attributed to the synergistic effect of CF and nano-Fe_3O_4.

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ABSTRACT

Ferroferric oxide (Fe_3O_4) nanoparticles were introduced into carbon fiber (CF)/cement composites to enhance the electromagnetic interference (EMI) shielding effectiveness (SE) in X-band (8.2–12.4 GHz). The spherical Fe_3O_4 nanoparticles were synthesized through solvothermal method, whose average diameter was about 30–50 nm. It was observed that incorporation of Fe_3O_4 nanoparticles (5 wt%) along with 0.4 wt% CF in the cement matrix led to a shielding effectiveness of 29.8 dB, which had 34.4% increase than that of CF/cement composite. The excellent EMI shielding property was attributed to the synergistic effect between CF and Fe_3O_4 nanoparticles. SEM revealed the homogeneous dispersion of Fe_3O_4 and CF in the cement matrix. The CF/Fe_3O_4/cement composite is believed to be a promising material for high EMI shielding.

1. Introduction

Over recent decades, with the popularity of electronic and communication equipment, electromagnetic wave technology has rapidly developed in many fields, such as military, commerce and industry [1]. While it offers conveniences to consumers, it also brings the problems of electromagnetic interference (EMI) pollution and information security, which are harmful to the personal health and military secrets [2]. Hence, in order to alleviate the electromagnetic radiation pollution problems, it is imperative to develop high frequency electromagnetic shielding materials which act as a barrier to restrict the admittance of electromagnetic wave by reflecting or absorbing [3].

As one of traditional building basic materials, cement has been widely used in construction field. Developing cement-based electromagnetic shielding composites is conducive to large-scale electromagnetic protection [4]. But cement mortar has poor electromagnetic shielding effectiveness, therefore, conductive or magnetic fillers are developed as EMI shielding materials in cement-based composites, such as carbon fiber (CF) [5-8], steel fiber [9], carbon black [10], carbon nanotube (CNT) [3,11,12], and ferrite [13] et al. The excellent properties of CF [14-16] like high strength, high modulus and good electrical conductivity make it the most promising filler for fabricating high-performance EMI shielding composites. D.D.L. Chung's group studied the application of CF, coke, graphite, carbon filament in cement based electromagnetic shielding materials and expounded the mechanism of electromagnetic shielding systematically [5,17-19]. They found that the electromagnetic interference shielding effectiveness of cement mortar reached 10 dB with 0.5 wt% CF and 4 mm of thickness in the frequency range of 1.0–2.0 GHz. Wang [6] et al. investigated the effect of freezing-thawing cycles on the electromagnetic wave reflectivity of CF/cement composites. With the increase of CF, the porosity of the composites decreased after freezing-thawing cycles and the reflectivity improved obviously. Cao [7] and his research team prepared the composites with short CF. The complex permit-

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tivity was observed in the temperature range from 30 °Cto 600 °C in the X-band and the shielding effectiveness was greater than 10 dB in the investigated region. However, the CF surface is inert and so is difficult to disperse in cement matrix, which further has direct effect on the shielding effectiveness. In order to promote the dispersion of CF in cement, efforts have been made on the CF surface treatment. In our previous work [8], with the thickness of 10 mm the cement-based composite modified with graphene oxide-deposited CF showed high shielding effectiveness up to 34 dB in the frequency band of 8.2–12.4 GHz (X-band).

On the other hand, many researches on EMI shielding effectiveness of nanocomposites have been conducted during the past decade [20,21]. Nano-Fe_3O_4 is the most popular magnetic material, which has been successfully prepared and shown remarkable electromagnetic interference shielding [22,23]. It possesses many merits, such as a high surface energy, large specific surface area, and satisfactory soft magnetization [24,25]. Generally, Fe_3O_4 nanoparticles were combined with conductive material, such as CF, CNT or graphene oxide (GO) in order to broaden the width of the frequency range of electromagnetic shielding. Liu [26] et al. reported the EMI SE exceeded 100 dB in the frequency of 3.22–40 GHz for tri-layer composites with nano-Fe_3O_4, nano-Fe and CNTs. M. Bayat [27] et al. studied the EMI shielding efficiency of hybrid multifunctional Fe_3O_4/carbon nanofiber composite in the frequency range of 8.2–12.4 GHz. The SE was influenced by contents of Fe_3O_4 nanoparticles (wt%) as well as pyrolysis temperature and shield thickness. They acquired the SE of as high as 70 dB with the dominant absorption mechanism due to the effect of both highly electrically conductive carbon nanofiber and the presence of Fe_3O_4 as magnetically permeable. Moreover, researchers have studied the synergetic interactions between magnetic nanoparticles and conductive fiber. Crespo [28] and his coworkers investigated the synergistic effect of the Fe_3O_4 nanoparticles and carbon nanofibres on electromagnetic shielding. The results showed that the synergistic effect notably increased the permittivity, AC conductivity and electromagnetic shielding efficiency. However, there are few reports about exploring CF and Fe_3O_4 nanoparticles synergy effect on electromagnetic interference shielding effectiveness in cement matrix. In this work, Fe_3O_4 nanoparticles were prepared by solvothermal method, which were mixed with CF and cement to produce cement-based composites. The aim of this study was to investigate the electromagnetic shielding effectiveness of the cement-based composites which was affected by CF and Fe_3O_4 nanoparticles. For this purpose, four different types of samples with different mass fractions of Fe_3O_4 nanoparticles were prapared. The electromagnetic shielding effectiveness (SE) of Fe_3O_4/CF/cement composites in the X-band frequency region (8.2–12.4 GHz) was investigated.

2. Experimental

2.1. Materials

Portland cement 42.5 was used as the matrix. The sand used met ISO standard (GB/T17671-1999) which was purchased from Xiamen ISO standard sand Co., Ltd. The CF T700SC was supplied by Toray Company, whose average diameter was about 7–8 μm. Hexahydrated ferric chloride ($FeCl_3{\cdot}6H_2O$), ethylene glycol ($(CH_2OH)_2$), ammonia hydroxide ($NH_3{\cdot}H_2O$), polyvinyl pyrrolidone (PVP) and carboxymethyl cellulose were of analytical grade and purchased from Aladdin Industrial Corporation.

2.2. Preparation of Fe_3O_4 nanoparticles

The solvothermal process was applied to prepare Fe_3O_4 nanoparticles. 19.5 g $FeCl_3{\cdot}6H_2O$ was dissolved in 360 mL ethylene glycol solvent by the aid of the magnetic stirrer in advance. Then 18 mL ammonium hydroxide and 1.2 g polyvinyl pyrrolidone were added in turn. The mixture was transferred into the sealed Teflon-lined stainless-steel autoclave and kept for 24 h at 180 °C. After the reactor cooled to room temperature, poured out the supernatant and rinsed the black solid product 3 times with deionized water and anhydrous ethanol in order, respectively. Finally, the Fe_3O_4 nanoparticles were obtained.

2.3. Mixing procedure and sample preparation

CF was cut to 3–5 mm manually and dispersed in water with the carboxymethyl cellulose through ultrasonic treatment. Carboxymethyl cellulose was used as primary dispersant in the amount of 1% by weight of water. The water/cement/sand/CF ratio by mass of the specimens was selected as 48/100/100/0.4, and the Fe_3O_4 nanoparticles were incorporated at concentrations of 1%, 3% and 5% by mass of cement (The volume fractions of CF and 5wt.% Fe_3O_4 nanoparticles were 0.18% and 0.76%, respectively). To ensure workability of the admixture, 0.5 wt% water reducing agent and 0.02 wt% defoaming agent were added. The mortar mixtures were prepared in a mortar mixer. The mixtures were mixed and cast in silicone molds. After 24 h, the specimens were removed from the molds and transferred to a moist-curing room before EMI SE testing.

2.4. Characterizations

The surface topography of CF/Fe_3O_4/cement composites was characterized by SEM (FEI QUANTA FEG 250 fieldemission SEM system). XRD (D8 ADVANCE, Bruker, Germany) was employed to analyze the structure of Fe_3O_4 nanoparticles. The magnetic properties of Fe_3O_4 nanoparticles were investigated by a vibrating sample magnetometer (VSM, Model 7400 series, Lakeshore). The composites specimens were made in sizes 22.4 × 10 × 7 mm for measuring the EMI properties in the frequency range of 8.2–12.4 GHz (X-band) by Agilent vector Network Analyzer (Agilent N5234A). At least 6 specimens were prepared for each kind of composites. The final results were averaged from all the tested samples.

3. Results and discussion

3.1. Characterizations of Fe_3O_4 nanoparticles

The crystal structure of the Fe_3O_4 nanoparticles was determined with XRD. As shown in Fig. 1, the characteristic diffraction peaks of Fe_3O_4 nanoparticles, 2θ = 30.1°, 35.5°, 43.2°, 53.5°, 57.1° and 62.7° correspond to (220), (311), (400), (422), (511) and (440) planes of Fe_3O_4, which are indexed to a face-centred cubic structure of magnetite (JCPDS19-0629) [29,30], respectively. Therefore, it can be concluded that the Fe_3O_4 nanoparticles were successfully synthesized.

The morphology of Fe_3O_4 nanoparticles was characterized by SEM (Fig. 2). As can be seen, the Fe_3O_4 nanoparticles have relatively uniform spherical morphology and the average diameter of the Fe_3O_4 is measured to be about 30–50 nm. The Fe_3O_4 nanoparticles stack closely with each other owing to their fine grain and high surface energy.

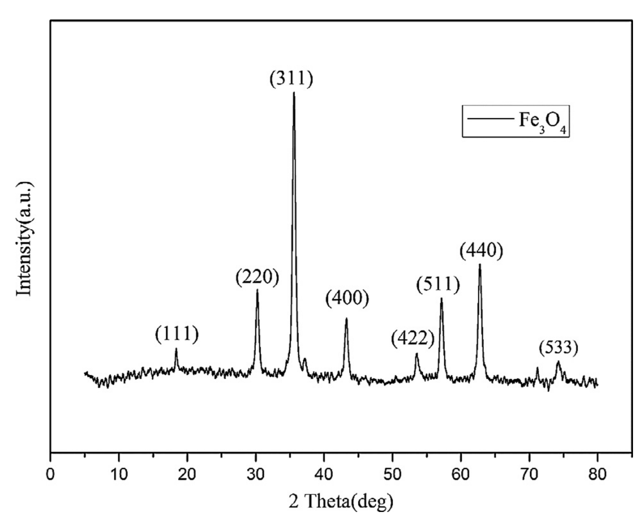

Fig. 1. XRD diffraction pattern of Fe_3O_4 nanoparticles.