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Microstructure, electrical conductivity and microwave absorption properties of γ-FeNi decorated carbon nanotube composites

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ABSTRACT

Nano-composite particles of γ-FeNi alloy/carbon nanotube (γ-FeNi/CNT) were successfully synthesized by wet chemical method. Microstructure of the as obtained nanoparticles was characterized by XRD, TEM, and EDS. The particles were then dispersed into epoxy resin to make conductive and microwave absorbing composites. Results have shown that the expected γ-FeNi nanoparticles were uniformly deposited a on the surfaces of CNTs with an average diameter of 16 nm. The epoxy based nanocomposites exhibited semi-conductive behavior and a maximum reflection loss of −15.4 dB at 16.5 GHz were obtained with thin thickness (1.6 mm) and low particles loading (2 wt%) of specimen. With similar microwave absorption strength and thickness of specimen, the particles loading used in this research (2 wt %) was much more lower than that reported in previous studies (~30 wt%).

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1. Introduction

Carbon nanotubes (CNTs) are a unique class of nanomaterial that can be applied to a wide range of fields due to its outstanding mechanical [1], thermal [2] and conductive properties [3,4]. Large specific surface area and tubular structure of CNTs provide an interesting scope for hosting a variety of materials. Thus, CNTs have been decorated with various materials, such as metals, alloys, compounds, molecules and even biomolecules, creating a new family of nanocomposites that combine the functionality of different materials into one [5–7]. One of the most promising and popular research interests is the metal-modified CNTs composite nanoparticles which have potential applications in the areas of high density data storage, biomedical fields, microwave absorption and shielding, etc. [8–10]. When the CNTs are attached with second phase nanostructures on the surfaces or encapsulated with them inside the cavities of the tubes, the composites may exhibit ideal electromagnetic absorbing properties [11,12]. Electromagnetic (EM) wave absorption properties of ferromagnetic alloys have been proved to be better than that of pure metal or metal oxides [13]. Among various alloys, FeNi alloy is widely used in applications such as EM wave absorbers, antennas, magnetic sensors and actuators, catalysts, and magnetic recording systems due to high EM properties and stable structure. Meanwhile, CNTs have been proved to be good dielectric loss absorbers, whereas the relatively low permeability limits potential applications in EM-absorbing materials. Therefore, nanocomposites of FeNi alloy modified CNTs will be a potential wave-absorbing materials with broad bandwidth and strong absorption due to high dielectric loss and magnetic loss [14].

The most common methods used to prepare metal or metal alloy modified CNTs are known as wet chemical technique (WCT), arc-discharge method (ADM), template-assisted method (TAM) and pyrolysis of organometallic precursor (POP). WCT involves pretreatment of CNTs, solution reaction, calcination and reduction reaction [5,9,15,16]. This method is simple and various metal or metal alloys and other nano-phases can be attached on or encapsulated into the CNTs [9,15,16]. But tube damage during pretreatment and diameter selection of CNTs are the drawbacks [9]. In situ filling metals or metal carbides into the CNTs by using ADM is time effective [9,17]. However, requirements for high temperature, low filling percentage, and unexpected metal carbides are the main shortcomings [9,17]. TAM has played an important role in the field of nanomaterials. Highly aligned metal-filled CNTs with high filling percentages and controllable size can be obtained by using this convenient method. Nevertheless the shell structure of the synthesized CNTs is poor and thus limits its application [18,19]. POP is

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widely used due to extensive organometallic precursors, high purity of products, and convenient operations. However, some organometallic precursors are toxic and many surface defects occur on the synthesized CNTs [19]. From afore-mentioned methods, it can be concluded that wet chemical method is preferred from viewpoints of economy, environment, and efficiency.

To date, some efforts have been carried out to prepare FeNi/CNT nanoparticles and comprehensively discussed the functions of the products. Wang et al. [12] used constant current electrodeposition method to fill CNTs with FeNi alloy, and the products exhibited good magnetization and electrocatalytic activity even with a low filling rate. Wu et al. [20,21] modified CNTs with FeNi alloy via wet chemistry method and crystalline FeNi was formed, results showed FeNi can be filled into the tubes or coated on the surfaces of CNTs. Lv et al. [22–24] filled CNTs with FeNi alloy nanowires and the microstructure, properties and application of the composite nanoparticles were comprehensively studied, results have shown that a reflection loss of −15 to −10 dB in the frequency range 10–16 GHz could be obtained with nanoparticles at a loading of 30 wt% with specimens having a thickness of 1.6–2.5 mm. The γ-FeNi alloy nanoparticles have been encapsulated in CNTs by sol–gel fabrication and hydrogen reduction method by Xu et al. [25] reported, complex permittivity, complex permeability, and microwave absorption features was measured, yielding a maximum reflection loss of −5 dB for a specimen with thickness of 1.5–2 mm. A recent paper by Wen et al. [26] investigated the synthesis and microwave absorption properties of FeNi/CNT nanoparticles by wet chemical method, and relatively good absorption of −20 dB from 2.72 to 18 GHz was obtained with high loading of FeNi/CNT (40 wt %) and thicker specimens. It can be clearly seen from the limited literature that the hotspots are usually investigations of microstructure and magnetic property of the FeNi/CNT nanoparticles [12,19–23]. Its potential applications in the fields of conductors or microwave absorbing materials have rarely been addressed [24–26]. Some studies showed good microwave absorption properties of the FeNi/CNT modified composites but with high loading of nanoparticles, which would make the processing more complicated [24,26].

Among various crystal structures of FeNi alloy, the γ-FeNi is relatively stable and exhibits a weak oxidation even storing for a long time [25]. The goal of this study is to prepare γ-FeNi alloy nanoparticles modified CNTs and evaluate the functionality of the nanoparticles acting as electrical conductor or microwave absorber with less weight loading and thinner thickness of specimens. Wet chemical method was used to prepare the composite nanoparticles, and the microstructure, conductivity and microwave-absorbing property of the obtained composites were comprehensively investigated.

2. Experimental

2.1. Materials

The multi-walled CNTs (MWCNTs, purchased from Chengdu Organic Chemicals Co.) used in this study had an outer diameter of 30–50 nm and length between 10 and 20 μm. The MWCNTs were functionalized with hydroxyl (−OH) at a weight ratio of 0.71%. Other reactants and reagents, including $Fe(NO_3)_3 \cdot 9H_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, KOH, ethanol and ethyl acetate, were obtained as analytical grade (supplied by Chinasun Specialty Products Co.). A low-viscosity epoxy resin LY 1564 and corresponding curing agent Aradur 3486 (supplied by Huntsman) were used to prepare nanoparticles filled composites. Ethyl acetate and ester dispersant (Chengdu Organic Chemicals Co.) were used to crash the agglomerates and reduce the viscosity in the mixing step for following composites preparation.

2.2. Preparation

The hydroxyl modified CNTs were firstly washed several times with distilled water and dried at 400 °C for 30 min to remove trace impurities before performing preparation. Then, the purified CNTs were dispersed in distilled water at a ratio of 1 g/L by ultrasonic agitation. $Fe(NO_3)_3 \cdot 9H_2O$ and $Ni(NO_3)_2 \cdot 6H_2O$, at atomic ratio of 2:3, were added into the CNTs solution and sonicated at 60 °C for 1.5 h. The ratios of CNTs to ferric nitrate and nickel nitrate were set as 1:0.004 (g: mol) and 1:0.006 (g: mol), respectively. Subsequently, a certain amount of KOH was dissolved into the solution to maintain its pH value at 11 within the ultrasonic bath for 0.5 h, and then hydroxide precipitate was formed. The solution was filtered and the obtained precipitates were dried in a vacuum oven at 80 °C. The precipitates were ground to form superfine powder, by calcinated at 400 °C for 2 h under an argon atmosphere, and (the metal oxide) reduced under argon/hydrogen atmosphere for 4 h at 600 °C. The as obtained sample was cooled down to room temperature and identified as containing γ-FeNi/CNT nanoparticles.

The electrical conductivity and microwave absorption properties of the as prepared γ-FeNi/CNT nanoparticles homogeneously dispersed within epoxy resin have been investigated. The functionalized composite material was labeled as γ-FeNi/CNT/EP. For comparison, the CNTs filled epoxy (CNT/EP) and neat epoxy (EP) specimens were also prepared. The weight ratio of the CNTs to epoxy, and γ-FeNi/CNT to epoxy were both varied from 0 to 4 wt% to find an appropriate content, respectively. The nanoparticles were firstly mixed with ethyl acetate and ester dispersant, and ultrasonically dispersed for 0.5 h. Then, the epoxy resin was added into the mixtures. The mixtures were planetary stirred (LY100, Atouch) every other 3 min for 1 h. The curing agents were finally added into the mixtures, at a weight ratio to epoxy of 34:100. The samples were slightly stirred and then degassed, casted in a mold, cured at 100 °C for 5 h. The dimensions of the specimens were 205 mm × 165 mm × 1.60 mm.

2.3. Characterization

The phase composition of the prepared nanoparticles was analyzed by X-ray diffraction (XRD, Bruker D8 Advance, Germany) with Cu Kα radiation at a scanning rate of 2°/min in the 2θ range from 20° to 100°. The morphology of the products was characterized by transmission electron microscopy (TEM, JEM2011) with accelerating voltages of 200 KV. Simultaneously, elemental analysis on the nanoparticles was done by the Energy-dispersive X-ray spectrometry (EDS) spectroscope attached to the TEM machine. Diameters of γ-FeNi nanoparticles within the TEM photograph were measured using software ImageJ. To evaluate the dispersion of CNTs and γ-FeNi/CNT particles in the epoxy resin, fractured surfaces of some typical specimens with various weight loadings of nanoparticles were observed with a SEM (Philips XL 30 ESEM instrument).

Electrical conductivity of the specimens was measured using a four-probe method (Loresta GP resistance meter). The conductivities obtained from this method are direct current (DC) conductivities, which can be calculated using the measured resistance and the geometry of the samples (according to standard of SEMI MF 84-1105). The effective alternating-current (AC) conductivities of the specimens with appropriate content of CNTs and γ-FeNi/CNT particles were measured using an Agilent 4294A impedance analyzer in the frequency range of 100 Hz–110 MHz with an AC voltage of 500 mV. This process was carried out by using of parallel plate

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