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Preparation and microwave shielding property of silver-coated carbonyl iron powder

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ABSTRACT

Electroless silver coating of carbonyl iron powder is demonstrated in the present investigation. The carbonyl iron powders are characterized by scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDX), and X-ray diffraction analysis (XRD) before and after the coating process. The relatively uniform and continuous silver coating is obtained under the given coating conditions. In this paper, the electromagnetic interference (EMI) shielding mechanism of the silver-coated carbonyl iron powder is suggested. The reflection of silver coating and absorption of carbonyl iron powder dominate the shielding mechanism of the silver-coated carbonyl iron powder. The silver-coated carbonyl iron powders are used as conductive filler in electroconductive adhesive for electromagnetic interference shielding applications. The effect of the thickness of electroconductive adhesive on the shielding effectiveness (SE) is investigated. The results indicate that the SE increases obviously with the increase of the thickness of electroconductive adhesive. The SE of the electroconductive adhesive with 0.35 mm thickness is above 38 dB across the tested frequency range.

1. Introduction

With the development of electronic information technology and widespread use of electronic devices, electromagnetic interference (EMI) has become a serious problem in recent years. The EMI may lead to malfunctioning of sensitive electronic components/appliances or may exert harmful effect on organisms. To solve this problem, a number of electromagnetic wave shielding materials, such as polymeric composites filled with carbon nanotubes, nickel coated carbon fibers, and silver coated cenospheres or copper powders, have been intensively investigated [1–4].

The primary mechanism of EMI shielding is usually reflection. The electromagnetic wave reflecting materials must have mobile charge carriers (electrons or holes), which possessing high conductivity. The conducting compositions based on metals (e.g. silver or copper powder), carbon materials (e.g. graphite, carbon black, carbon fibers or carbon nanotubes) and core–shell particles (e.g. silver coated cenosphere, copper or mica composites) are commonly used for EMI shielding applications [3–16]. The secondary mechanism is absorption which requires the presence of dielectric or magnetic dipoles (e.g. $BaTiO_3$, Fe_3O_4, carbonyl iron powders, Fe-filled carbon nanotubes, Co/SiO_2 nanosphere or Ni/cenosphere core–shell composites) [17–23].

Carbonyl iron powders are widely filled in resin or rubber as electromagnetic wave absorbers due to their low price, high specific saturated magnetization, and Snoek's limit [24–26]. He and Guan [27] mixed the spherical and flaky carbonyl iron powders to adjust effectively the electromagnetic parameters and enhance the absorption property in high frequency. Tong [28] mixed carbon nanotubes/carbonyl iron powders (CNTs/CIPs) composite with different mixture ratios to enhance the microwave absorption properties by a mechanically milling method. Compared with CIPs, the CNTs/CIPs composites had higher electrical conductivity, permittivity, and dielectric loss, which gradually increased with the increasing CNTs content. Silver is a typical EMI shielding material with high SE. The core–shell structured composites with carbonyl iron cores and silver shells may generate a high SE due to the EMW reflection of silver coating and the absorption of carbonyl iron powder. Therefore, the development of conductive–magnetic composite materials with composition and well-defined structures is valuable for excellent EMW shielding capabilities. However, a few investigations have been done on the core–shell composite fillers with magnetic cores and conductive shells. Fe_3O_4/Ag core–shell composite nanoparticles were synthesized by an easy processing route [29]. The composite nanoparticles exhibited typical superparamagnetic behavior, and high conductivity. Gong [30] prepared

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the Fe_3O_4/Ag nanoparticles with both superparamagnetic and antibacterial properties by reducing silver nitrate on the surface of Fe_3O_4 nanoparticles using the water-in-oil microemulsion method. And Garcia-Torres [31] synthesized silver-coated cobalt (Co/Ag) nanoparticles by a micellar method.

In the present work, silver-coated carbonyl iron powders were prepared by an electroless plating method. Electroconductive adhesive was obtained by mixing epoxy resin with silver-coated carbonyl iron powder. The EMI shielding mechanism of the silver-coated carbonyl iron powder was discussed. And the effect of the thickness of electroconductive adhesive on the SE was investigated in terms of the packing structure of the silver-coated carbonyl iron powders.

2. Experimental

2.1. Materials and chemicals

Carbonyl iron powders used in the present investigation were supplied by Guangzhou Yuelong Metal Powder Co., Ltd., China. Epoxy resin (E-44) was purchased from Guangzhou Shenghao Chemical Industry Co., Ltd., China. The chemicals used for the electroless Ag-coating of carbonyl iron powders included $AgNO_3$ (99.9+%), HCHO (37 wt.%), C_2H_5OH (99.9%), $C_2H_8N_2$ (⩾98%), and NH_4OH (28.0–30.0% NH_3), which were of analytical grade and purchased from Guangzhou Chemical Regents Company. Deionized water was used throughout the work.

2.2. Electroless plating procedures

Ammonia as complexing reagent was added dropwise to the $AgNO_3$ (0.05 mol/L) solution, and adjusted the mixed solution pH value to 9.5. At the conditions, all Ag^+ formed complex compound $[Ag(NH_3)_2]^+$ with NH_3. A total of 4 g of carbonyl iron powders were stirred in the silver ammonia solution (300 mL), as solution A. HCHO (4 mL) was dissolved in 100 mL of ethanol, as solution B. The solution A and B were preheated to 50 °C. After that, solution B was added to solution A, and stirred for 20 min. The silver-coated particles were then filtered off, washed with deionized water, and dried at 100 °C for 2 h.

2.3. Preparation of adhesives and samples

The electrically conductive adhesives were prepared by mixing epoxy resin, silver-coated carbonyl iron powders and anhydrous ethanol used as solvent at the mixing speed 1000 rpm using a lab-scale homogenizer (FJ-200) for 15 min. The homogenizer was manufactured by Changsha Qiulong Instrument and Equipment Co., Ltd., China and the speed range was 50–10,000 r/min. Then ethylenediamine used as hardener was mixed, and the mass ratio of ethylenediamine to epoxy resin was 1:10. The mass ratio of silver-coated carbonyl iron powders to epoxy resin was 7:3, and the mass ratio of anhydrous ethanol to epoxy resin was 6:10. The single overlapped adhesive joints used for studying the EMI shielding behavior were prepared with bonding of PTEF (polytetrafluoroethylene, Yangcheng Electric Model Co., Ltd., China) film with ∅115 mm and 2 mm thickness. The adhesive joints were hardened at 50 °C for 10 h.

2.4. Characterization

The morphology of the uncoated and silver-coated carbonyl iron powders were characterized by SEM (S-3400N(II), Hitachi). The structure analysis of the powders was carried out using XRD (Ultima IV, Rigaku) with Cu Kα1 radiation.

2.5. Measurements of SE

The SE of the sample for plane-wave conditions/simulation was measured by means of the coaxial cable method [32]. The setup consisted of a DN15115 SE tester, which was connected to an Agilent 4396B RF network spectrum impedance analyzer. The scanning frequency ranged from 100 kHz to 1.5 GHz. The thickness of the adhesive layers were 0.2 and 0.35 mm.

3. Results and discussions

Fig. 1 shows the XRD patterns of (a) carbonyl iron powders and (b) silver-coated carbonyl iron powders. The XRD pattern in Fig. 1(a) indicates that the microstructures of carbonyl iron powders are mainly composed of Fe comparison with the standard data of JCPDS (06-0696). As shown in Fig. 1(b), the diffraction peaks at 38.12°, 44.28°, 64.43°, 77.40° and 81.54° are clearly seen, besides

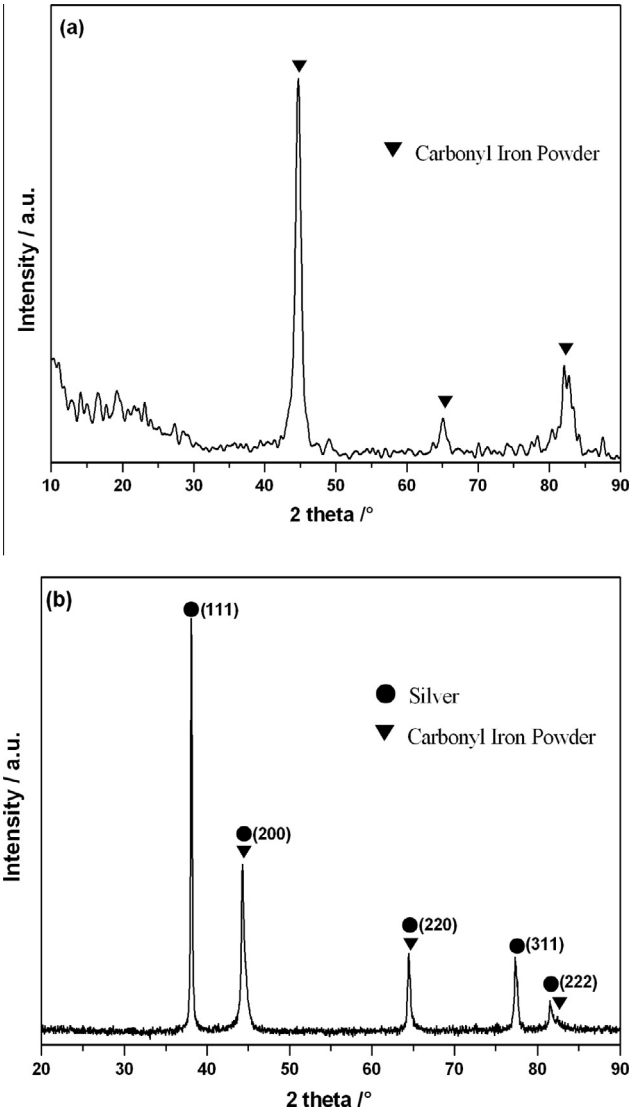

Fig. 1. XRD patterns of (a) carbonyl iron powders and (b) silver-coated carbonyl iron powders.

the weak diffraction peaks of carbonyl iron powders. The results are matched with the major peaks (111), (200), (220), (311) and (222) of Ag comparison with the standard data of JCPDS (04-0783), which reveals a pure silver layer successfully coated on the surface of carbonyl iron powders.

Fig. 2 shows the SEM micrographs of (a) carbonyl iron powders and (b) silver-coated carbonyl iron powders. As seen from Fig. 2(a), the carbonyl iron powders have a regular spherical surface morphology and are well dispersed from each other. As shown in Fig. 2(b), the uniform and compact coating is observed, where a few grains adhering on the surface of silver-coated carbonyl iron powders. It appears that during the electroless silver deposition, the uniform and compact silver coating develops through silver particles formation.

Fig. 3 shows the SEM micrographs of (a) cross-section and (b) surface of the electroconductive adhesive filled with silver-coated carbonyl iron powders. The silver-coated carbonyl iron powders as the conductive particles are surrounded by epoxy resin and uniformly distributed. The particles touch one another and form a more compact packing structure, which benefits for the electrical conductivity and SE of the electroconductive adhesive.