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Electrophoretic deposition of spherical carbonyl iron particles on carbon fibers as a microwave absorbent composite

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ABSTRACT

Carbonyl iron (CIPs) micron size spherical particles were deposited on carbon fibers by electrophoretic deposition method (EPD). A well stabilized suspension of CIPs was prepared in acetone-ethanol media and two different stabilizer of Iodine and Trietanolamine (TEA) were used to investigate stability of suspension. X-ray Diffractometer (XRD) was employed to study structure of CIPs, CFs and CFs/CIPs composite. Field Emission Scanning Electron Microscopy (FE-SEM) images were taken to see the morphology of CFs and deposited particles. Dynamic Light Scattering (DLS) and zeta potential test were carried out to study the stability of particles and also to see the average particle size of powder. To investigate magnetic and reflection loss properties, Vibrating Sample Magnetometer (VSM) and Vector Network Analyzer (VNA) were used, respectively. Uniformly coated fibers were selected to make composites and results revealed that the best Reflection Loss (RL) was related to CFs coated for 20 minutes in a bath containing iodine with a layer thickness of 2 mm. Maximum recorded RL was found to be -13.8 dB at frequency of 9.22 GHz.

1. Introduction

The trend to use microwave absorbing materials in the area of computers [1], network equipment [2], telecommunication [3], data transfer and information technology [4], automotive electronics [5] aerospace and defense [6] and electromagnetic interference shielding (EMI) [7] has been growing extensively. Materials having these properties are divided into three groups: (1) dielectric attenuated (2) magnetic attenuated and (3) combination of them [8–10]. To satisfy the requirements of a microwave absorbing composite, two conditions must be fulfilled. First, the impedance of the absorbing material should match the free space impedance; second, the incident electromagnetic wave should be attenuated while passing through the absorbing layer [11]. A good absorber should be light weight, wide band, thin in thickness and mechanically strong [12]. Most of the recent research works have tried to meet these requirements using different magnetic materials like CIPs. CIPs based absorbers have been widely studied by researchers due to their higher saturation magnetization (compared to ferrites) and higher Curie temperature [13]. However, relying on magnetic materials as the only constituent of an absorber, has not shown promising results. Therefore, many studies have focused on the carbon based additives such as reduced graphene oxide (RGO) [14], carbon nanotubes (CNTs) [15], multi-walled carbon nanotubes (MWCNTs) [16] and carbon fibers (CFs) [17,18]. Over the last decade, many methods have been proposed by researchers to fabricate the CIPs based composites with superior absorptive properties. For instance, Lv et al. synthesized hierarchical hollow carbon/Fe/Fe3O4 nanospheres by a simple template method and subsequent pyrolysis process with a tunable thickness of hollow carbon spheres [19]. In another study conducted by the same group, porous coin-like iron with an excellent absorption property of −53.8 dB at frequency of 16 GHz was achieved [20]. However, a controllable and versatile method is demanding to tailor the properties of these composites structurally in either nano or micron scale. Thus, we propose the electrophoretic method to deposit CIPs on CFs to fabricate a microwave absorbing composite. EPD is easy to set up and handle, cost effective, controllable and versatile [21]. The aim of our study is to control the morphology of electrophoretically deposited CIPs on CFs and also obtaining a proper RL over the frequency range of 8 to 12 GHz.

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2. Experimental

2.1. Materials

Pure iodine (99.85%), acetone (99%) and ethanol (96%) supplied by Alpha Chemika in India, hydrochloric acid (98%) and Triethanolamine (2922 13 10 Merck) supplied by Merck, gray spherical CIPs with a mean particle size of 2 μm supplied by Chengdu Punda Metal Material Co, Ltd, carbon fibers (D = 0.7 μm) supplied by Chongqing Joywell Trade Co, China Ltd, Epoxy (Viscosity in 25 °C = 1.75 MPa.S and specific weight 1.15 g/cm^3) and its hardener (Viscosity in 25 °C = 1.1 MPa.S and specific weight= 1.0 g/cm^3) were all purchased and used without further purification.

2.2. Preparation of suspension

Two suspensions were prepared from 100 cc acetone-ethanol (A-E) organic solution. The ratio of acetone/ethanol was set to be 1:1. First solution was stabilized using five drops of TEA and its pH was adjusted to be 3 using oleic acid. Second solution was prepared using 0.03 g iodine as a stabilizing agent and the pH was again set to be 3 but this time by 2 M hydrochloric acid. Subsequently, the prepared suspensions were sonicated for 15 minutes. Before EPD process, CFs were weighed by a balance with an accuracy of 0.001 g and then treated with 65w% nitric acid for 4 hours, washed with deionized water and dried at 120 °C for 24 hours to increase adherence between particles and CFs [22].

2.3. EPD process

For preparation of EPD setup and establishing the best EPD process, a cylindrical borosilicate glass beaker was used as the bath. A stainless steel electrode with dimensions of 2.5 × 10 cm and a bunch of pan based CFs with approximate dimensions of 2.5 × 10 cm were taken as anode and cathode, respectively. The distance between anode and cathode was adjusted to be 3 cm. Maximum effort was taken to set the CFs as much as parallel to stainless steel electrode to prevent non-uniformity of deposited particles. A power supply of DC with a maximum voltage of 45 V and 5A was employed for EPD process. Different deposition processes were conducted using suspensions of (E-A) solutions for both stabilizing agents. Iodine containing suspension was coded “SI” and TEA containing suspension was coded “ST” . To determine optimum voltage, firstly, time was taken constant and voltage was altered from 10 V to 30 V by a 5-V step for both “ST” and “SI” samples and each process was performed during 4 minutes. After determination of optimum voltage for proper deposits of CIPs, time parameter came to play the role and EPD was performed for 4, 8, 12, 16 and 20 minutes at the voltage of 30 V. CIPs /CFs, extracted from this step, were coded SI_4, SI_8, SI_{12}, SI_{16}, SI_{20}, and ST_4, ST_8, SIT_{12}, ST_{16}, ST_{20}. After Each EPD process, the CIPs/CFs were carefully extracted from suspensions and immersed in distilled water for several seconds in order to have loose particles segregated and prevent sudden evaporation of (E-A) at ambient temperature which in turn causes micro cracks on CIPs/CFs coating. Finally, the dried CFs/CIPs were weighed to find the mass ratio of deposited particles and CFs (CIPs: CFs).

2.4. Heat treatment of CIPs/CFs

In order to remove iodine and any organic agents from coating surface and also to understand its effect on microwave absorption, CIPs/CFs were heat treated in a simple electric furnace at temperatures of 100 and 200 °C with a heating rate of 1 °C/min and maintained at these temperatures for 2 hours.

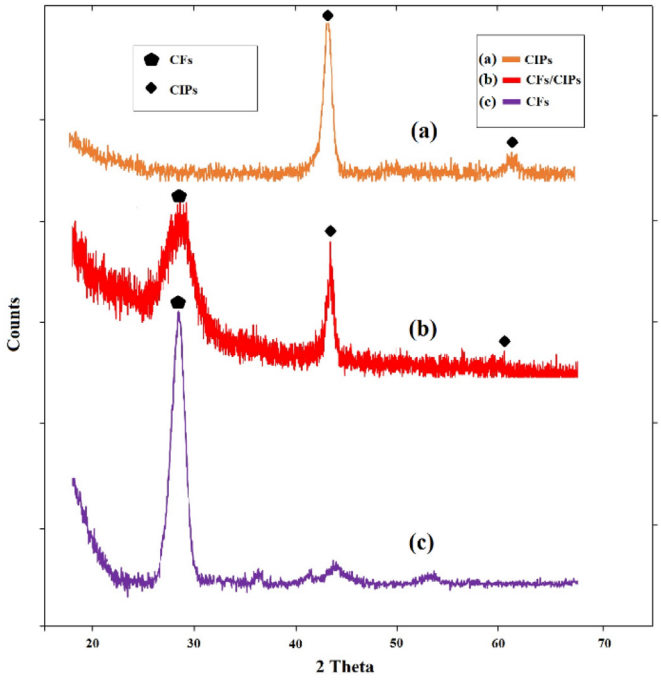

Fig. 1. XRD patterns of CIPs, CIPs/CFs and CFs.

2.5. Fabrication of CIPs/CFs composites

CIPs/CFs were cut carefully to the length of 2 mm. 0.15 g CIPs/CFs was mixed with 0.3 g epoxy and the mixture was then vibrated slowly until become homogenous. An aluminum substrate with dimensions of 2 × 1 was employed and before using, was dipped in a 70 w % sulfuric acid at temperature of 80 °C for 3 hours to increase the interfacial bonding strength between epoxy and substrate. The epoxy and CIPs/CFs mixture was slowly laid on substrate in a way that completely covered the surface in which the thickness of coating was adjusted to be 1 and 2 mm. Finally, two drops of hardener was added to the composition.

2.6. Materials characterization

XRD of the CIPs was studied using a Brucker AXF (D8 Advance) with a CuKa radiation source (λ = 0.154056 nm) generated at 40 kV and 35 mA under the angle of 10 to 90°. FEG-SEM images were taken (by MIRA3 FEG-SEM, Tescan, Czech and by S-4160 FEG-SEM, Hitachi, Japan) to investigate morphology of deposition. DLS analysis (by Nanotrac Wave, Microtrac, USA) was used to determine the particle size distribution and Zeta potential of the suspension. The microwave absorption properties and RL of CIPs/CFs composites measured over the frequency range of 8–12 GHz by Vector Network Analyzer (VNA model HP) at room temperature. Magnetic properties of CIPs/CFs composites were studied with a VSM (C300) at room temperature.

3. Results and discussion

3.1. Structural analysis

Fig. 1 shows XRD patterns of CIPs (a), CF/CIPs (b) and CFs (c). Corresponding JCPDS cards for the peaks existing in the patterns are No. 01–0640 and No. 06–0696 for (a) and (c), respectively. A diffraction peak at 2θ = 26.347° and two other diffraction peaks at 2θ = 52.379° and 65.02° can be assigned to scattering from the (002), (110) and (2 0 0) planes of CFs and CIPs crystal lattice, respectively. Pattern (b) demonstrates that CIPs particles exist on CFs.