Journal of Colloid and Interface Science 533 (2019) 297–303

Contents lists available at ScienceDirect

Journal of Colloid and Interface Science

journal homepage: www.elsevier.com/locate/jcis

Regular Article

Cobalt nanoparticles embedded nitrogen-doped porous graphitized carbon composites with enhanced microwave absorption performance

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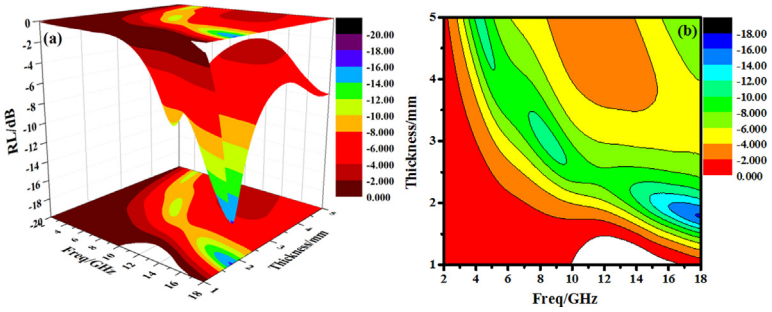
GRAPHICAL ABSTRACT

The as-prepared Co@N-C composites exhibit great microwave absorption performance, which is derived from the strong dielectric attenuation, multiple microwave scattering and dielectric polarization, as well as shortened impedance matching.



ARTICLE INFO

Article history:
Received 29 July 2018
Revised 24 August 2018
Accepted 24 August 2018
Available online 25 August 2018

Keywords:
Graphitized carbon
Cobalt nanoparticles
Porous
Microwave absorption
Impedance matching

ABSTRACT

For high-efficiency microwave absorption, both of the self loss of materials (dielectric loss and magnetic dissipation) and structural attenuation (multiple scattering, interfacial polarization) play important roles. In addition, the magnetic/dielectric materials combination, and void volume introduction can also contribute to the optimization of impedance matching. Given that, 2D cobalt nanoparticles embedded nitrogen-doped porous graphitized carbon composites (Co@N-C) were fabricated via a simple sacrificial templates method, where the CoAl-layered double hydroxide (CoAl-LDH) nanosheets were prepared to hold ZIF-67 and then decomposed during the sintering process. In this work, strong dielectric attenuation, multiple microwave scattering and dielectric polarization, as well as shortened impedance matching all make for the nice microwave absorption performance. This work not only exhibits the importance in materials selection and structure design, but also demonstrates the close relation between matching thickness and response frequency at maximum reflection loss (*RL*) peak.

1. Introduction

The widely used electronic and communication devices in civil field cause growing electromagnetic (EM) interference, which has threatened the health of human being and normal operation of electronic equipment [1–3]. Microwave absorbing materials are identified as promising candidates to solve this issue by converting EM wave into thermal energy via different loss mechanisms, avoiding the secondary contaminations [4,5].

A rational design of microwave absorber should not only consider the intrinsic properties of selected materials, but also

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https://doi.org/10.1016/j.jcis.2018.08.083
0021-9797/

optimize the architecture to achieve the EM wave absorbing as much as possible [6]. According to the impedance matching condition $Z_{in} = Z_0(\mu_r/\varepsilon_r)^{1/2}$[7], a strong magnetic stuff combined with a moderate dielectric ingredient is a suitable match [8]. Only in this way will the impedance matching and dissipation ability could be both ensured. In terms of the structural design, 2D laminated nanostructure and porous feature are both ideal alternatives [9,10]. 2D nanosheets often possess special electrical behavior, large specific surface area and low mass density due to their confined electron-strong interaction in a plane, large anisotropy and quantum effect. In addition, given that nanosheets can be easily fabricated into high-orientation multilayered composites, they can act as a good barrier to energy wave by increasing the propagation path [11,12]. As for the porous feature, the void stacked volume can induce more interfacial polarization and microwave scattering, meanwhile the air inside the pores could also shorten the impedance gap between air and absorbers [13].

Based on the above analysis, we tried to fabricate a laminated magnetic/dielectric composite with porous structures. ZIF-67 was firstly coated onto the surface of CoAl-LDH nanosheet template, followed by a sintering process. During the thermal treatment period, ZIF-67 was decomposed into cobalt particles embedded graphitized carbon, and honeycomb holes were obtained after the pyrolysis of ZIF-67. Moreover, the final sample still remains the original flake-like morphology as CoAl-LDH template. Due to the 2D flake structure, nanoporous feature, magnetic/dielectric combination, the as-prepared composites exhibit good microwave absorption property. In addition, the relationship between matching thickness and response frequency of maximum absorbing ability was further detected.

2. Experimental

2.1. Synthesis of CoAl-LDH templates

Typical method was used to prepare the CoAl-LDH Templates. In brief, a given mass of $CoCl_2{\cdot}6H_2O$, 0.61 g of $AlCl_3{\cdot}6H_2O$, and 1.06 g of urea were added into three-neck flask with 40 mL of deionized water. The obtained solution was refluxed under continuous stirring at 95 °C for 2 days. Finally, powder products were obtained through filtration with water and ethanol for several times and subsequent drying at vacuum oven.

2.2. Synthesis of CoAl-LDH@ZIF-67 precursors

0.1 M $Co(NO_3)_2{\cdot}6H_2O$ was added into 50 mL of methanol solution dissolved with 0.1 g as-prepared CoAl-LDH, then 50 mL of 0.8 M 2-methylimidazole (2-MeIM) was added into the above solution with mild stirring. The product was gained after centrifugation with methanol and drying in vacuum oven.

2.3. Synthesis of ZIF-67

ZIF-67 was prepared as the same method above. Typically, Co $(NO_3)_2{\cdot}6H_2O$ was added into 50 mL of methanol to get solution A, 2-methylimidazole was added into 50 mL of methanol to get solution B. Mixed solution was obtained after the mixture of solution A and solution B. The ZIF-67 powder was gained after centrifugation with methanol and drying in vacuum oven.

2.4. Synthesis of Co@N – C composites

As-prepared CoAl-LDH@ZIF-67 was calcinated at 800 °C for 2 h under N_2 atmosphere. The product was got after centrifugation with ethanol and water, as well as drying in vacuum oven, which is denoted as Co@N–C Composites.

2.5. Characterization

The as-prepared samples were extensively characterized by scanning electron microscope (SEM), X-ray diffractometer (XRD), transmission electron microscopy equipped with energy-dispersive X-ray spectrometer (TEM-EDS), Raman spectroscopy, BET, X-ray photoelectron spectroscopy and so on.

2.6. Electromagnetic parameter measurement

Electromagnetic parameters were detected using Agilent PNA N5244A vector network analyze. The mixture was previously synthesized by homogeneously mixing wax (30 wt%) and samples, then compacted them into toroidal-shaped samples (Φ_{in}: 3.04 mm, Φ_{out}: 7.0 mm).

3. Results and discussion

In order to demonstrate the successful preparation of target samples, X-ray diffraction technique was use to detect the phase characters. As shown in Fig. 1, the XRD patterns of CoAl-LDH@ZIF-67 contain both the diffraction peaks characters of CoAl-LDH and ZIF-67, exhibiting the successful preparation of CoAl-LDH@ZIF-67 precursors. Thermal treatment was conducted to obtain the Co@N–C Composites, which was also detected by XRD test. As can be seen from Fig. 1, three evident diffraction peaks corresponded to (1 1 0) (2 0 0) (2 2 0) planes can be observed, which is well consistent with the standard PDF cards of Cobalt. In addition, the stability of Co@N-C was also detected. As seen in Fig. S2, the XRD pattern of Co@N-C exposed in heating oven at 50 °C for 3 days (Co@N-C-T) exhibits similar crystal structure as

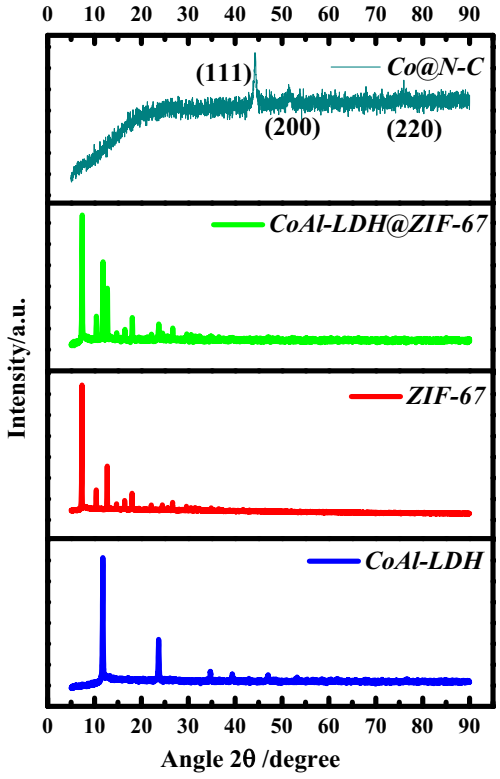

Fig. 1. XRD patterns of CoAl-LDH, ZIF-67, CoAl-LDH@ZIF-67 and Co@N-C.