Journal of Alloys and Compounds 509 (2011) 494–498

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

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

Synthesis and microwave absorbing properties of FeCo alloy particles/graphite nanoflake composites

Chen Wang[a], Ruitao Lv[a], Zhenghong Huang[a], Feiyu Kang[a,b,*], Jialin Gu[a]

[a] State Key Laboratory of Fine Ceramics and New Process, Department of Materials Science and Engineering, Tsinghua University, Beijing 100084, China
[b] Advanced Materials Institute, Graduate School at Shenzhen, Tsinghua University, Shenzhen, Guangdong Province 518055, China

ARTICLE INFO

Article history:
Received 31 March 2010
Received in revised form
12 September 2010
Accepted 18 September 2010
Available online 24 September 2010

Keywords:
FeCo alloy
Graphite nanoflake
Microwave absorption
Soft magnetism
Co-deposition

ABSTRACT

Exfoliated graphite was processed into nano-sized flakes by jet milling, ultrasonication and acid treatments, and then FeCo alloy particles were uniformly deposited onto the surface of the prepared graphite nanoflakes (GNFs) by a co-deposition and subsequent annealing process in reducing atmosphere. The FeCo-GNFs composites demonstrate good soft magnetic performance and effective microwave absorption, especially in the lower frequency range. The strongest absorption reaches −30.6 dB at 7.4 GHz with a coating thickness of 2 mm. Furthermore, effective microwave absorption at different frequency bands can be acquired by adjusting the coating thicknesses and filling ratios of FeCo-GNFs absorber. Considering the low cost and high efficiency, the FeCo-GNFs composites have great potential as microwave absorber for practical applications.

1. Introduction

The requirements for an ideal microwave absorbing material (MAM) include thin coating thickness, low density, broad absorbing band and strong absorption. The usual radar frequency band locates at 2–18 GHz and it is usually difficult to obtain an effective absorption in the lower frequency range, especially in 2–4 GHz with thin coating thickness. In this range, some kinds of ferrites and Fe-based materials have demonstrated great absorbing performance [1–7]. However, they still have some drawbacks, such as high density and requiring high mass filling percentages, which are usually over 60 wt%, or even 90 wt%. Compared to ferrites and Fe-based materials, graphite powder possesses much lower density and stronger electric loss. Furthermore, if graphite particles can be separated into nanoflakes with platelet shape, more effective absorption might be obtained due to the advantages of platelet-shaped materials than the rod-shaped and sphere-shaped ones in microwave absorption applications [8]. Therefore, GNFs are promising as one kind of low density electric loss absorber for MAMs. However, GNFs do not show the magnetic loss for microwave, which limits its applications in this field due to their thicker coatings and higher absorbing frequency band. For instance, Lee et al. [9] synthesized graphite nanoplates/epoxy composites and showed absorbing peaks around 15 GHz. However, the coating thicknesses were more than 7 mm. Fan et al. [10] treated flake graphite by ball milling and ultrasonic stirring in ethanol solution of phenol-formaldehyde cement (EFC). The coating thickness of flake graphite/EFC was only 2 mm and the absorption was less than −25 dB and the absorbing peak still located at higher frequency band (over 14 GHz). In order to obtain a thinner coating (e.g. 2 mm) with effective microwave absorption at the same time, it is necessary to functionalize the GNFs to enhance their magnetic properties. Depositing magnetic metal particles onto the surface of GNFs may be a feasible route. Fan et al. [11] coated graphite with Ni by an electroless plating method. The absorbing peaks located at frequency range of 8–18 GHz and the absorption was not very strong. Liu et al. [12] and Yang et al. [13] used the GIC method to synthesize Fe/graphite and FeNi/graphite materials respectively. The lower frequency absorption of graphite was improved remarkably. However the absorption was not strong enough (above −7 dB with 2 mm thickness) and the process was so complicated. We also synthesized FeCoNi/graphite absorber [14], which possessed the strongest absorption of −24 dB and the absorbing peak was still in higher frequency range (over 12.2 GHz).

FeCo alloy is one kind of excellent soft magnetic material. Recently, the studies on synthesis and applications of FeCo alloy nanoparticles and nanowires have been initiated because

* Corresponding author at: Room 2714, Yifu Building, Tsinghua University, Beijing 100084, China. Tel.: +86 10 62773752; fax: +86 10 62771160.
E-mail address: fykang@tsinghua.edu.cn (F. Kang).

0925-8388/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.jallcom.2010.09.078

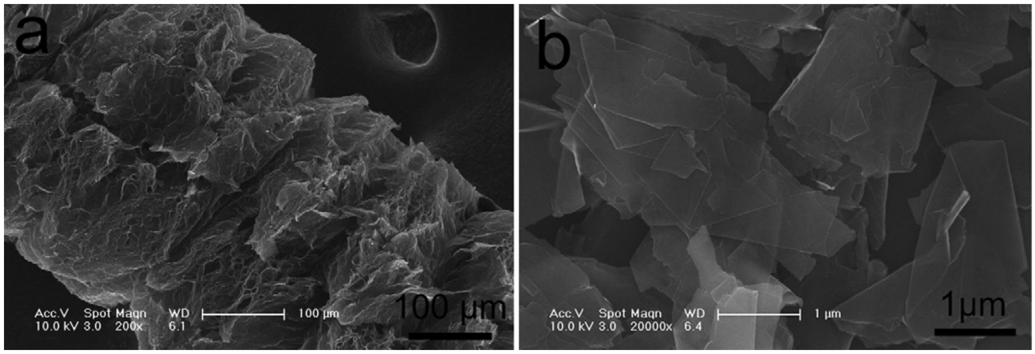

Fig. 1. SEM images of graphite samples. (a) Original exfoliated graphite with a worm-like structure, (b) as-prepared GNFs after a series of post-treatments.

of their microwave absorbing performance [6,7,15–19]. Nie et al. [6] synthesized FeCo alloy particle absorber by mechanical alloying process, which had an excellent absorption in 2–4 GHz. Yang et al. [7] synthesized FeCo nanoplates by reducing aqueous Fe^{2+} and Co^{2+} with hydrazine, and the absorber showed great absorption in different frequency range by controlling the coating thickness. These studies indicate that FeCo may be a good material to be deposited on GNFs. In the present work, a traditional co-deposition method, which is commonly used in synthesis of ferrite materials, has been improved. We use this simple and effective method to load FeCo alloy particles onto GNFs and design a novel microwave absorber, which can combine the excellent electric property of GNFs and magnetic property of FeCo alloys together, and may lead to an ideal microwave absorbing performance especially in the lower frequency range.

2. Experimental

2.1. Sample preparation

The exfoliated graphite was firstly treated by jet milling and then ultrasonic stirring in isopropanol for 2 h. After that, the sample was treated in a hot mixing acid solution (HNO_3:H_2SO_4 = 1:1) for 4 h. Subsequently, the as-treated exfoliated graphite was put into a solution containing different molar ratios of $FeSO_4$ and $CoSO_4$ (e.g. Fe:Co = 2:1, 1:1 or 1:2). The molar ratio of FeCo and GNFs is 1:1. NaOH solution was used to adjust the pH value of the solution to pH 12. The precipitate was separated and then annealed in reducing atmosphere (Ar/H_2) for 1 h at 300 °C, 450 °C and 600 °C, respectively. Finally, the products were cooled to room temperature in Ar ambient to obtain FeCo-GNFs samples. The pure FeCo alloy powder (Fe:Co = 2:1, 450 °C annealed) was also synthesized by the same method for comparison.

2.2. Characterization

X-ray diffraction (XRD, D/max-2500) was used for phase analysis. Scanning electron microscope (SEM, FEI SIRION 200) and transmission electron microscope (TEM,

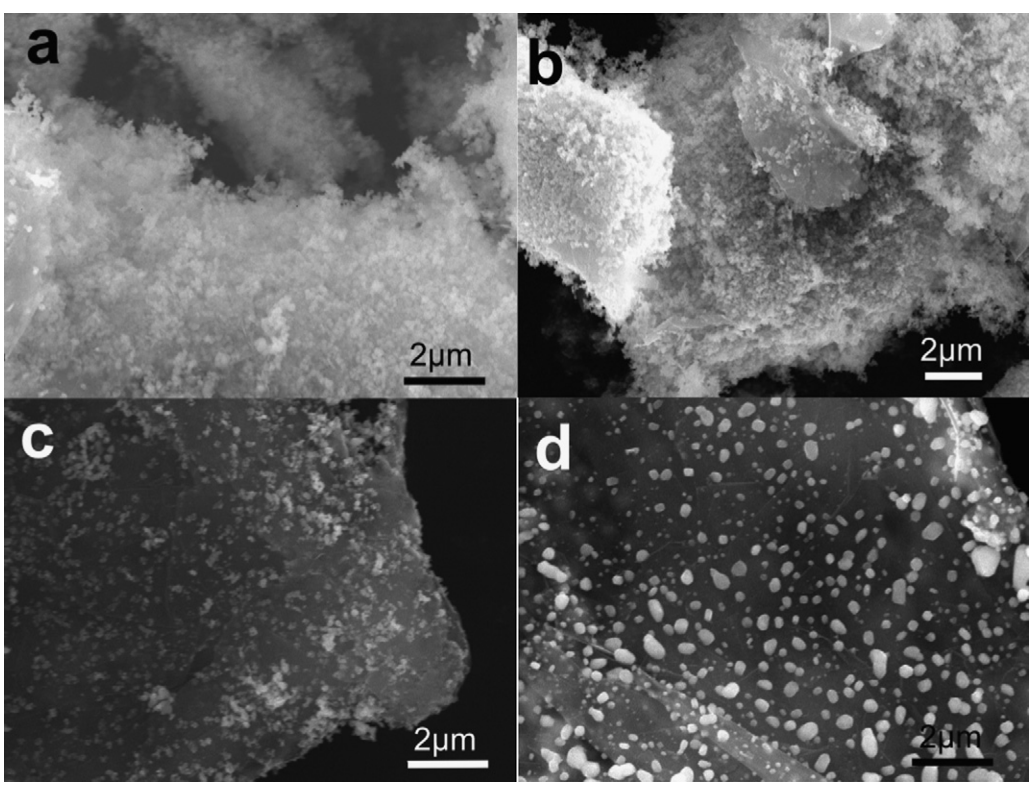

Fig. 2. SEM images of the different FeCo-GNFs samples with the ratio of Fe:Co = 2:1. (a) Untreated sample and samples annealed at (b) 300 °C (c) 450 °C (d) 600 °C in H_2 atmosphere, respectively.