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Review

Synthesis, structure and anti-oxidation properties of FeNi nanoparticles coated by BN (hexagonal)

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ABSTRACT

BN-coated FeNi nanoparticles (i.e., FeNi/BN) has been prepared via liquid phase reduction process followed by high temperature calcination. TEM and SEM images confirm the nano-dimensional structure with core/shell structure, with FeNi NPs as core and BN layers as shell. The anti-oxidation performance of the coated and uncoated powders were investigated from room temperature to 900 °C by TGA and DTA, thermal analysis data revealed that the FeNi NPs are oxidized at 220 °C in the air, while the FeNi/BN samples are stable below 570 °C. This results from the fact that the enhancement of the thermal stability by the BN shell. In addition, the BN layers limit the growth of FeNi NPs, resulting in the lower saturation magnetization and higher coercivity, compared with pure FeNi NPs.

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1. Introduction

Traditional soft magnetic materials of FeNi NPs are different from Fe and Ni NPs with high permeability, surface magnetism and low energy loss have been widely used in electromagnetic wave absorber, high density magnetic recording materials, high effective catalyst and diagnostics agents [1–6]. Unfortunately, oxidation and wear of the metal surfaces are fatal flaws when the size of the alloy is decreased to nanometer level, and the oxidation would actually worsen the magnetic properties of alloy [7].

One way to solve these drawbacks is to coat soft-magnetic metallic nanoparticles with an inorganic coating to build the core/shell nanostructure, such as silicon dioxide [8], graphite [9] and boron nitride [10]. The coating structure not only transforms

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the magnetic properties of the alloy, but also improves the anti-oxidation of the alloy. Hexagonal boron nitride (h-BN) has received considerable attention due to its high thermal stability, electrical conductivity, low dielectric constant and mechanical strength [11,12], widely used in gaseous uptake, protective coatings, advanced ceramic composites and catalyst support [13–19]. Therefore, the h-BN layers are ideal shells because they can protect metal nanoparticles effectively against oxidation and limit the growth of the alloy.

Several process, including magetron, ion-beam co-sputtering [20] and spraying methods [21] have been developed to prepare BN coated nanoparticles. However, these methods still exit all sorts of drawbacks such as complicated working procedure and difficulty in microstructure control. It has been urgent affairs to produce a large number of nanocapsules with good property.

The purpose of the present work is threefold. The first was to develop a new and simple method for producing the FeNi/BN samples, therefore, the mixture of FeNi alloy precursors and H_3BO_3 powders were selected. The second purpose was to investigate the microstructure of FeNi NPs and FeNi/BN samples by means of X-ray powder diffraction, Transmission electron microscopy and Field emission scanning electron microcopy. The last was to compare the oxidation temperature of pure FeNi NPs and FeNi/BN, these characterization data will provide us with guideline for researching the BN-coated nanostructure materials.

2. Experimental procedures

2.1. Materials

Hexagonal boron nitride (h-BN) powders were synthesized via a simple high-temperature approach (900 °C). Iron nitrate hexahydrate, nickel nitrate hexahydrate, sodium borohydride, polyvinylpyrrolidone and ethanol were used in the experiment are analytically pure grade. Deionized water (DI water) is produced by a water purification machine.

2.2. Preparation of FeNi Nps

Firstly, 1.16 g nickel nitrate hexahydrate and 0.404 g iron nitrate hexahydrate were dissolved into a beaker with 60 ml alcohol for mixing. Then, adding 1.6 g PVP-K30 and 0.6 g sodium borohydride under stirring until dissolved, the black precipitate were obtained and washed to neutral by deionized water and ethanol for several times before dried at 60 °C for 6 h in a vacuum. The dried mixture (i.e., FeNi alloy precursors) was annealed with flowing ammonia at 900 °C for 2 h to obtain FeNi NPs.

2.3. Preparation of FeNi/BN

The FeNi alloy precursors and H_3BO_3 powders with molar ratio of 4:1 were well mixed by triturator. Then, the mixture powders were placed in an alumina tube mounted in a tube furnace and heated up to a temperature of 900 °C with flowing ammonia for 2 h, and naturally cooled (6 h) to room temperature to give a black precipitate.

2.4. Characterization

The phases formed in the FeNi NPs and FeNi/BN were characterized by X-ray diffraction (XRD, Japan, Rigaku smartlab) having Cu-Kα radiation ($\lambda = 0.15405$ Å) at a scanning rate of 8° per second in the 2θ range from 10° to 90°. Chemical bonding and groups were determined by fourier transform infrared spectroscopy (FTIR, Vector 22, Germany) between 500 and 4000 cm^{-1}. The morphology and microstructure of samples were investigated by transmission electron microscopy (TEM, Tecnai G2 F20, Netherlands) and field emission scanning electron microscopy (SEM, FEI Company, USA) with an energy dispersive analysis of X-rays (EDAX). The oxidation mechanism of samples were measured by thermal gravimetric analysis (TGA) and scanning differential thermal analysis (SDTA) during heating up from room temperature to 900 °C with 5 °C/min in air. The magnetic properties of FeNi NPs and FeNi/BN were evaluated by vibrating sample magnetometer (VSM, 7404, America) run under 20000 Oe field.

3. Results and discussion

3.1. Crystal structure and functional groups analysis

Fig. 1 illustrates the XRD patterns of the (a) FeNi NPs, (b) h-BN powders and (c) FeNi/BN. The characterization diffraction peaks at 44.30°, 51.56°and 75.90° are assigned to the (111), (200) and (220) crystallographic plants of FeNi NPs (Fig. 1(a)). As exhibited in Fig. 1(b), the peaks located at 25.86°, 41.78° and 76.32° matched well the (002), (100) and (110) reflections of the crystalline plants of h-BN [22–24]. After BN coated on the surface of FeNi NPs, as Fig. 1(c) shown, it is obviously observed that the characterization peaks of FeNi/BN are in good accordance with the bragg diffractions of FeNi NPs and h-BN powders, indicating that FeNi NPs can be relatively well combined with the h-BN. In addition, no peaks of metal hydroxides and metal oxides could be found throughout out the reaction and the good crystallinity of the samples were shown by strong peak intensity and narrow peak width, demonstrating that the purity of products were high and without oxidated, due to the better reducibility of ammonia gas and the protection of boron nitride layers.

Fig. 2 shows the FT-IR spectrum of (a) h-BN and (b) FeNi/BN. As is shown in Fig. 2(b), FeNi/BN exhibits three main distinct peaks of B—N—B, B—N and B—NH_2/B—OH bending at 785 cm^{-1}, 1383 cm^{-1} and 3451 cm^{-1}, respectively. The high frequency peaked at 3451 cm^{-1} is a typical —OH stretching vibration from surface atmosphere species [25]. Both of three peaks can be found in two samples, predicating the existing of BN phase in the sample. It should be noted that, the relative peak intensity of B-N-B and B-N bending vibration are decreased significantly compared with h-BN powders. It could be attributed to the presence of BN coating. Moreover, the h-BN presents additional peaks at 1080 cm^{-1} and

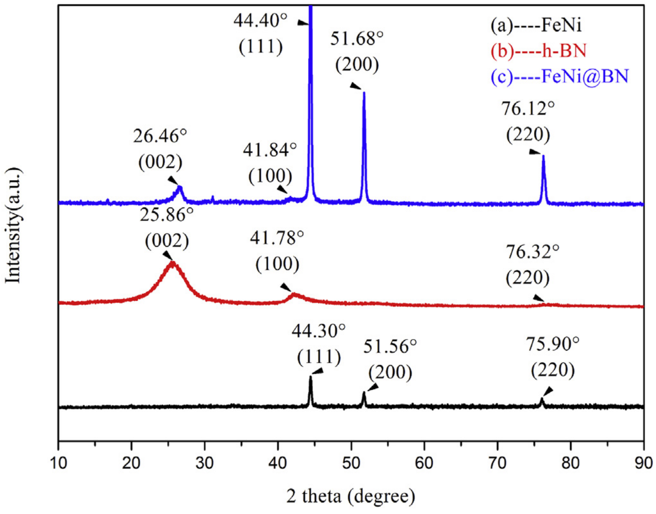

Fig. 1. XRD patterns of (a) FeNi NPs, (b) h-BN powders and (c) FeNi/BN.

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