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Decorating Mg/Fe oxide nanotubes with nitrogen-doped carbon nanotubes

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ABSTRACT

Mg/Fe oxide nanotubes decorated with nitrogen-doped carbon nanotubes (CN_x) were fabricated by catalytic chemical vapor deposition of ethylenediamine on the outer surface of oxide nanotubes. Mg/Fe oxide nanotubes were prepared using a 3:1 molar precursor solution of $Mg(NO_3)_2$ and $Fe(NO_3)_3$ and anodic aluminum oxide as the substrate. The obtained samples were characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS) and vibrating sample magnetometer (VSM). The XRD pattern shows that the oxide nanotubes are made up of MgO and Fe_2O_3. TEM and SEM observations indicate the oxide nanotubes are arrayed roughly parallel to each other, and the outer surface of oxide nanotubes are decorated with CN_x. XPS results show the nitrogen-doped level in CN_x is about 7.3 at.%. Magnetic measurements with VSM demonstrate the saturated magnetization, remanence and coercivity of oxide nanotubes are obvious improved after being decorated with CN_x.

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1. Introduction

Carbon nanotubes (CNTs) have been the focus of intensive study for their unique structural, electrical, and mechanical properties since their discovery by Iijima [1]. Their potential applications include nanodevices, quantum wires, ultrahigh-strength engineering fibers, sensors, and catalyst supports [2–4]. The doping of CNTs with nitrogen atoms is an effective way to tailor the electrical properties of CNTs. The additional electrons contributed by the N-doping provide electron carriers for the conduction band [5], which makes the N-doped CNTs (CN_x) either metallic or semiconductive with a narrow energy gap [6], thus offering the possibility of greater electrical conductivity than the pure CNTs.

Inspired by the discovery of CNTs, extensive research efforts have been devoted to tubular nanostructures. Various nanotubes, such as sulfides, nitrides, metals and oxides [7–10], have been synthesized in template matrices. Compared with other nanostructures, nanotubes possess low mass density, high porosity and increased surface area [11], which are especially important in applications involving interactions between gases or liquids and the surface of a material [12]. These nanotubes have made important contributions to fundamental research in the areas of catalysis [13], sensors [14], electronic or magnetic devices [15,16], and biological separation and transport [17].

To optimize the uses of nanotubes in many fields, it is necessary to decorate the surface of nanotubes. For example, decorating can improve dispersion of CNTs in solvents or impart new optical, electric, magnetic properties [18–20]. Recently, decorating CNTs with different materials, such as magnetic nanoparticles [21–23], noble metals [24–26], polymers [27–29] and biomaterials [30,31] have been extensively reported. However, there are few reports about decorating oxide nanotubes. In our previous work, Zn/Co/Fe oxide nanotubes [32] and CN_x [33] had been successfully prepared. Especially, CN_x were prepared with Mg/Fe oxides obtained from layered double hydroxides as catalysts. Basing on those works, we grow CN_x on the outer surface of Mg/Fe oxide nanotubes to obtain the decorated oxide nanotubes in this manuscript. We expect the decorated oxide nanotubes not only possess the excellent magnetic properties of magnetic oxide nanotubes, but also possess the greater electrical conductivity derived from CN_x.

2. Experimental

2.1. Preparation of Mg/Fe oxide nanotubes

Anodic aluminum oxide (AAO) membrane (Whatman, Anodisc 47, 200 nm) was commercially obtained and used as received. The precursor solution was prepared by dissolving $Mg(NO_3)_2$ and $Fe(NO_3)_3$ with a molar ratio of 3:1 (Mg:Fe = 3:1) in distilled water to form an aqueous solution of nitrate. Then the AAO template was immersed into the precursor solution for about 24 h at room temperature. Subsequently, the AAO template was put under vacuum at room temperature for 4 h and then dried at 100 °C for 4 h. After that, the template was heated at 500 °C for 1 h

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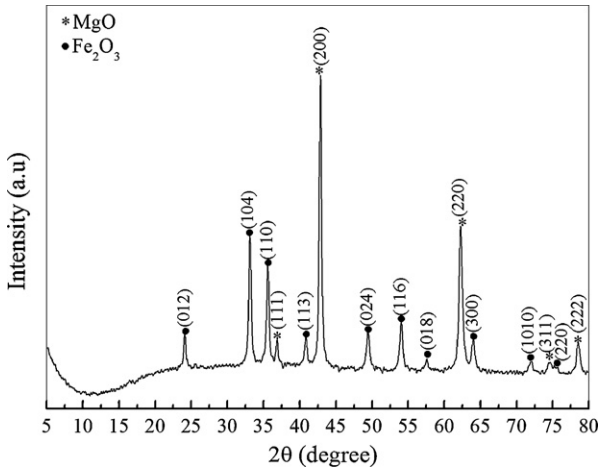

Fig. 1. XRD pattern of Mg/Fe oxide nanotubes.

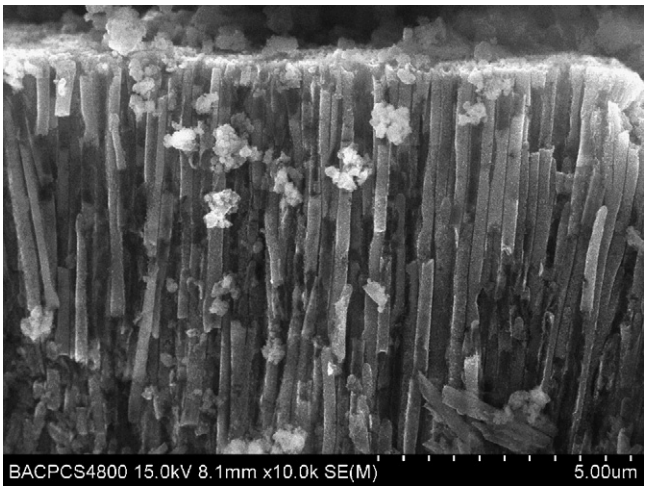

Fig. 2. SEM images of Mg/Fe oxide nanotube arrays.

in air, at a heating rate of 1 °C/min, and arrays of Mg/Fe oxide nanotubes inside the AAO templates were obtained. To remove the AAO template, they were then immersed in a 3 mol/L NaOH solution for 40 min at room temperature. Finally, after being washed with distilled water and dried at 100 °C for 2 h, one-dimensional Mg/Fe oxide nanotube arrays were obtained.

2.2. Preparation of Mg/Fe oxide nanotubes decorated with CN_x

About 20 mg of Mg/Fe oxide nanotubes were loaded in a quartz boat and placed in the middle of a tube furnace, which was heated under a flowing Ar (200 cm^3/min) at a rate of 1 °C/min. Between 350 and 400 °C, Ar was switched to H_2 (120 cm^3/min). On reaching 580 °C, ethylenediamine was pumped into the furnace at a rate of 0.15 cm^3/min with flow of Ar (400 cm^3/min). The reaction was maintained for about 20 min, after which the furnace was allowed to cool down to room temperature under Ar. Then the Mg/Fe oxide nanotubes decorated with CN_x were obtained.

2.3. Characterization

The X-ray powder diffraction (XRD) of oxide nanotubes was collected on an X'pert PRO MPD diffractometer operated at 40 kV and 40 mA with Cu Kα radiation. The morphologies of oxide nanotubes were observed through transmission electron microscopy (TEM) with a JEOL JEM-1200EX instrument operated at 100 kV. Field emission scanning electron microscopy (FE-SEM) was performed by using a

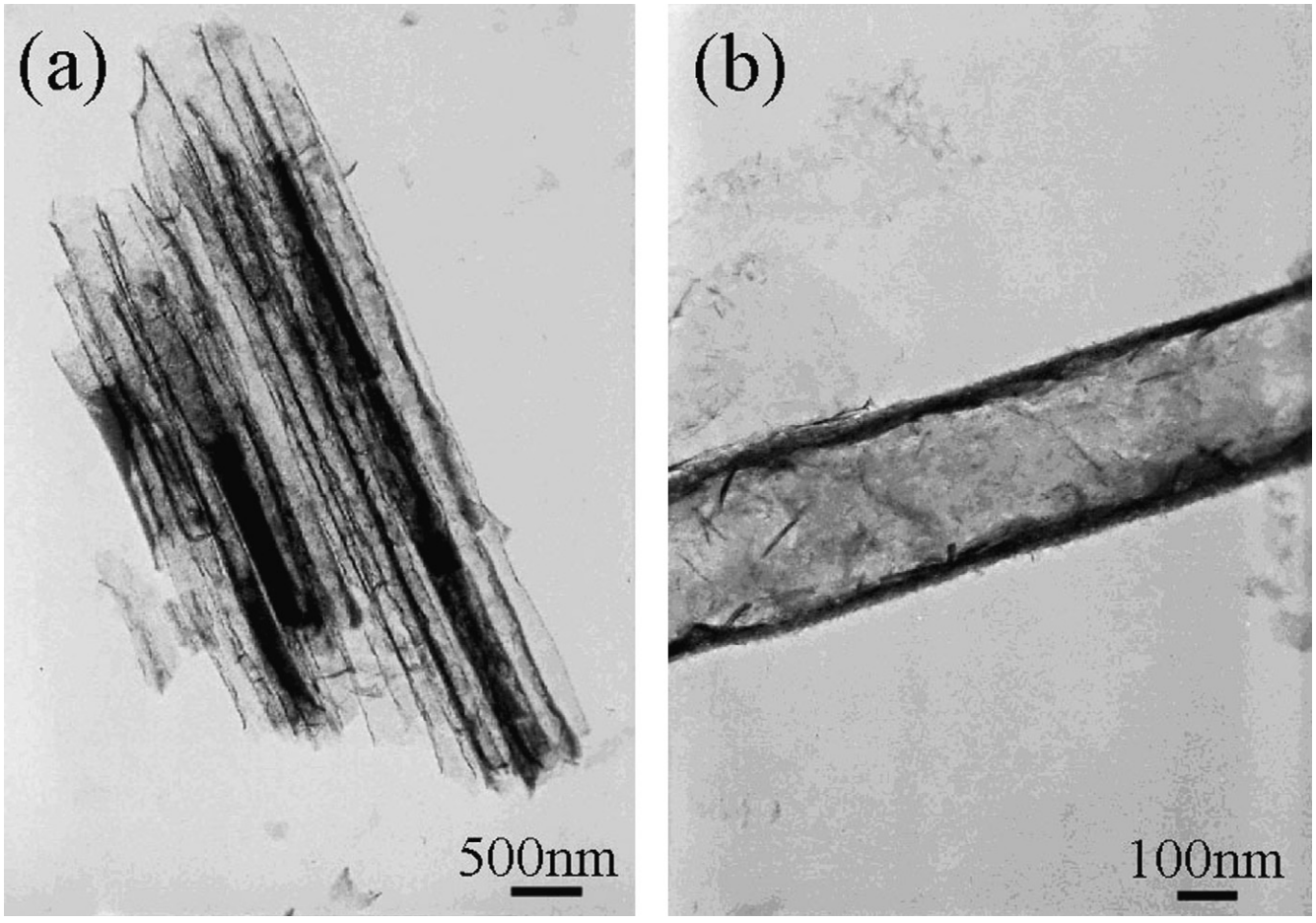

Fig. 3. TEM images of Mg/Fe oxide nanotubes.

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