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Synthesis and Characterization of FePt Nanoparticle/Single Walled Carbon Nanotube Composites

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Abstract. We report the synthesis and characterization of FePt nanoparticles (NPs)/single walled carbon nanotube (SWNTs) nanocomposites, which might have important potential applications to orient CNTs and the development of new catalysts. The crystallite size of FePt NPs is about 2.5-4 nm and the Fe atom content ranges from 50% to 65%. HiPCO SWNTs have been decorated with FePt nanoparticles under mild conditions without any pretreatment. When the bundle size of HiPCO SWNTs is about 20 nm, FePt NPs mostly attach on the sidewalls of SWNTs. The loading of FePt nanoparticles on the SWNTs bundles can be conveniently controlled by adjusting the initial amount of FePt NPs. The nanocomposite showed magnetic properties in a magnetic field. TEM, HRTEM, and EDS spectra were performed to observe the nanostructure of these nanocomposites.

Introduction

Carbon nanotubes (CNTs) have been intensively studied towards applications in many different fields for their unique mechanical, electronic, optical, and magnetic properties[1-3]. It is important for CNTs to be controlled by their chirality, separation, functional groups and orientation in the potential applications of CNTs, which are based on their electronic properties[4, 5]. Meanwhile, the nanocomposite of CNTs and metal nanoparticles have attracted many researches for their special catalytic properties, such as direct methanol fuel cell (DMFCs) applications[6]. Thus it is quite useful to obtain a nanocomposite to improve the CNTs catalytic and electrical properties, which could be composed by magnetic and catalytic metal nanoparticle and CNTs. Several methods have been reported to orientate CNTs in a magnetic filed[7, 8]. For example, Jia et al. used magnetic nanoparticles, such as Fe_3O_4 NPs, to attach onto CNTs to orient SWNTs in a magnetic field[7]. In catalytic research, Hsu et al. synthesized electrocatalyst, which is composed by the nanocomposite of RuPt nanoparticles and CNTs, to enhance the catalytic property for DMFCs[6]. Here, we report a novel nanocomposite of FePt NPs and single walled carbon nanotubes (SWNTs). Sun et al. firstly synthesized FePt NPs in 2000, which have attracted intensive studies for many applications such as high-density recording media and ferromagnetic nanocomposites[9]. Due to its high superparamagnetic property and the potential catalytic application, FePt NPs were chosen to synthesize the nanocomposite. In our work, SWNTs and FePt NPs have been prepared separately. Then we can control the FePt NPs decoration density by adjusting the initial ratio of FePt NPs to SWNTs.

Materials and methods

The SWNTs used in this work were purchased from Carbon Nanotechnologies Incorporation (CNI). Platinum (II) acetylacetonate [$Pt(acac)_2$, purity 97%], oleylamine, octyl ether and iron(III) ethoxide [$Fe(OEt)_3$, purity 95%]] were purchased from Aldrich. All solvents were of reagent grade quality and were used without further purification. Nitrogen gas (>99% purity) was purchased from Lixin Gas Corporation. A transmission electron microscope (TEM), a high-resolution transmission electron micrograph (HRTEM), and an energy dispersive X-ray spectroscopy (EDS) analysis were employed to study the SWNTs, FePt NPs and the nanocomposites in a JEM 2100F JEOL with an accelerating voltage of 200 kV.

The HiPCO SWNTs were used directly without any purification. According to the procedure of Saita and Maenosono[10], FePt NPs with crystallite sizes of 2.5-4 nm were synthesized under N_2 atmosphere at 298 °C. Then FePt NPs were dried at 60 °C overnight in an oven. 20 mg SWNTs and 20 mg FePt NPs were dispersed in 10 ml hexane by 15 minutes ultrasonication in a water bath, separately. Then the two reaction solutions were mixed and stirred at room temperature for 16 h. Finally, the mixture was centrifuged at 10,000 rpm for 15 min. After that, the black sediment was collected and denoted as FePt NPs/SWNTs nanocomposite. Then 1 mg nanocomposite was ultrasonicated for 15 min in 5 mL $CHCl_3$ in a water bath for TEM examination.

Results and Discussions

Fig.1 shows the TEM and HRTEM images of SWNTs, which were dispersed in $CHCl_3$. It is observed that the pristine SWNTs length is uniform and about several micrometers. The bundle size of SWNTs was about 20 nm, as seen from Fig. 1 (b), which was composed of about ten SWNTs.

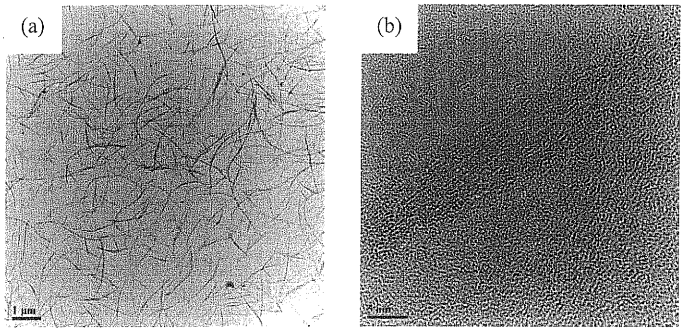


Fig. 1 TEM (a) and HRTEM images (b) of SWNTs dispersed in $CHCl_3$

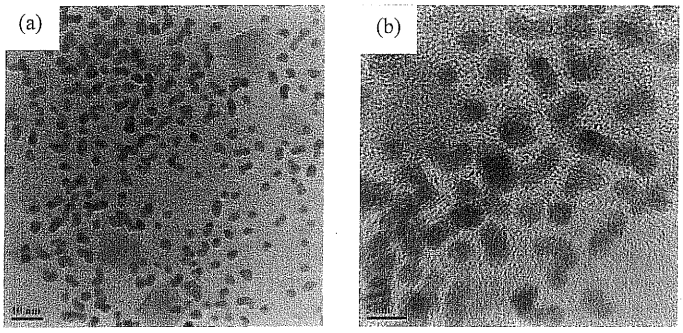


Fig. 2 TEM (a) and HRTEM images (b) of FePt NPs dispersed in hexane

Fig. 2 shows the TEM and HRTEM images of the prepared monodisperse FePt NPs, which was dispersed in hexane. As is shown in Fig. 2 (b), the crystallite size of FePt NPs is uniform with a narrow diameter range from 2.5 to 4 nm. The atomic content of Fe, shown in EDS spectra, ranges from 50% to 65%, which corresponds to an atom ratio of Fe to Pt ranging from 1 to 2.

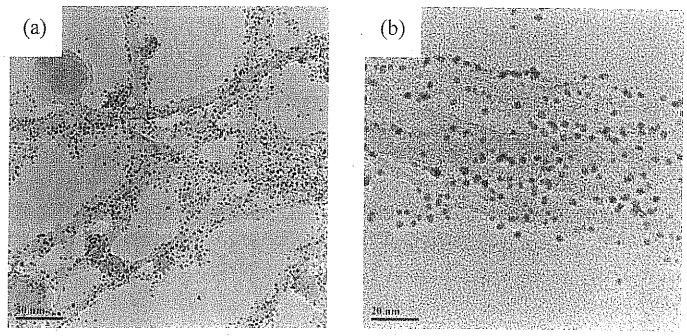
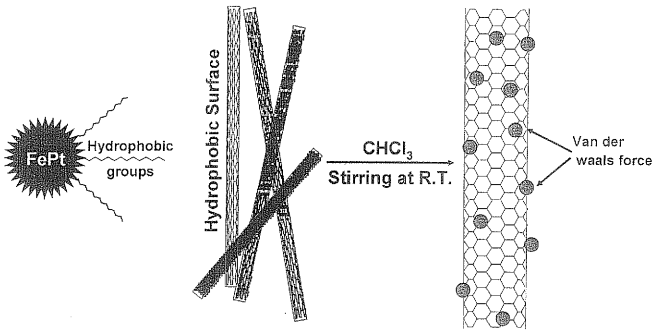


Fig. 3 TEM (a) and HRTEM images (b) of a FePt/SWNTs nanocomposite

Fig. 3 shows the TEM and HRTEM images of a FePt/SWNTs nanocomposite, which was dispersed in $CHCl_3$. Fig. 3 (a) shows that the FePt NPs are uniformly attached on the sidewalls of SWNTs bundle. When the diameter of SWNTs is over 20 nm, FePt mostly attach onto the sidewalls of SWNTs. Fig. 3 (b) is a HRTEM image of the FePt/SWNTs nanocomposite, displaying a layer-by-layer morphology of the sidewall of the SWNTs and the crystalline facets of FePt NPs, which demonstrate the highly crystalline nature of metallic FePt NPs. This indicates that the diameter ratio of SWNTs to FePt NPs play a key role during the assembly procedure. When the ratio is higher than 7, SWNTs can be easily decorated with FePt NPs during the assembly under mild experimental conditions. When the ratio is lower than 5, only a few FePt NPs can attach on the sidewalls of SWNTs. In addition, the amount of FePt NPs attached on SWNTs can be controlled by the concentration of FePt NPs, which will be discussed in another report.



Scheme. 1 Schematic Illustrations of the Assembly of FePt NPs on SWNTs

The possible synthesis mechanism of the FePt/SWNTs nanocomposite is schematically shown in Scheme. 1. Sun suggests that the surface of FePt NPs are generally stabilized with alkyl carboxylic acid (ROOH) and alkyl amine (RNH_2)[11]. Fe can be covalently linked by –COOH, forming iron carboxylate (–COO–Fe). And $–NH_2$ prefers to bind to Pt via a coordination bond. In our studies, the surfaces of FePt are stabilized by two single straight-chain hydrophobic groups ($-(CH_2)_{16}CH_3$ and $-(CH_2)_{17}CH_3$). Thus the surfaces of FePt NPs are coated with hydrophobic groups. As we know, the surface of SWNTs without a treatment is mainly graphite structure. In view of the inertness of SWNTs walls, we dispersed FePt NPs in $CHCl_3$, which is also an excellent solvent to wet SWNTs. The alkene groups prefer to interact with the SWNTs surfaces under stirring, namely, FePt NPs attach onto SWNTs through the hydrophobic segment. The hydrophobic interaction between the FePt NPs and the SWNTs made them tightly attached together. We also tested the magnetic

separability of the nanocomposite in $CHCl_3$, when placing a magnet near the glass bottle. The black sample was attracted toward the magnet in 1 h, which demonstrated its magnetic sensitivity and potential applications to orient CNTs.

Summary

In summary, a novel nanocomposite of FePt/SWNTs was synthesized and characterized. FePt NPs were successfully loaded on the sidewalls of SWNTs under mild experimental conditions and a possible formation mechanism was discussed. The FePt NPs/SWNTs nanocomposite can not only make oriented CNTs possible but also may have potential applications in catalysis and for sensors.

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