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Synthesis of novel single-walled carbon nanotube–magnesium nanoparticle composites by a solution reduction method

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ABSTRACT

Single-walled carbon nanotubes (SWNTs) reinforced with uniform distribution of magnesium nanoparticles were synthesized by a solution reduction method using a Grignard reagent and lithium naphthalide. Magnesium nanoparticles were observed to be of an average size of 4.5 ± 0.5 nm. Raman data indicated distinct diameter selectivity for larger-diameter metallic SWNTs of 2.21 nm and some degree of de-bundling of the nanotube bundles upon the incorporation of the magnesium nanoparticles.

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1. Introduction

Metallic nanoparticles are of particular interest because they often show size-dependent properties different from bulk matter [1,2]. In recent years, due to its unique lightweight properties, magnesium nanoparticles (Mg NPs) have emerged as a highly attractive candidate for a broad range of practical applications including batteries [3], automotive and armor composites, [4] and hydrogen storage [5]. There have been several reported synthetic strategies of Mg nanoparticles including electrochemical reduction [2,6], gas phase synthesis [7] and colloidal solution phase reduction [2,5]. To improve mechanical properties for high-strength low-density applications, researchers have synthesized Mg alloys and composites utilizing silicon [8], alumina [9] and carbon supports [10,11]. Despite the many advances in Mg incorporation techniques, there still exists the challenge of controlling the size, dimensions, and purity of these nanoparticles.

Specifically, carbon nanotube (CNT) is a highly attractive type of reinforcement phase due to its extremely high Young's modulus (up to 1 TPa) and strength (up to 150 GPa) [12]. Multi-walled carbon nanotubes (MWNTs) have been used to produce various metal-based composites with aluminum [13], titanium [14] and magnesium [15,16]. Gupta et al., [15] who used a powder metallurgy synthetic technique, reported increased thermal stability of Mg-MWNTs nanocomposites compared to monolithic pure Mg. To the best of our knowledge, the synthesis of Mg nanoparticles-single-walled carbon nanotubes (SWNTs) composites have not been reported and studied to date. Studies have shown that SWNTs offer superior performance, as compared with MWNTs, due to their smaller diameter and higher surface areas, and improved mechanical properties due to their highly crystalline structure [17].

Herein, an effective inexpensive technique of synthesizing SWNTs-Mg nanoparticle composites using a solution reduction method is described. Well-dispersed, uniform Mg nanoparticles (ranging 4.5 ± 0.5 nm in diameter) incorporated with SWNTs were observed. The SWNTs-Mg NPs were characterized by high-resolution transmission electron microscopy (HR-TEM), energy dispersive X-ray spectroscopy (EDS), and Raman spectroscopy.

2. Materials and experiments

Materials: The SWNTs (CoMoCat) with purity of $>90\%$ were obtained from SouthWest Nanotechnologies, Inc. Anhydrous tetrahydrofuran (THF), methylmagnesium chloride (3.0 M solution in THF), lithium, and naphthalene were purchased from Sigma-Aldrich. All reagents were used as received without further purification.

Synthesis of SWNTs-Mg NPs composites: All experiments were performed under an argon atmosphere. All glassware was dried in an oven overnight before use. Sample separation and preparation were performed under argon in a Captair Pyramid glove bag. Initially, 120 mg of SWNTs were sonicated in 40 mL of anhydrous THF in a 150 mL 3-neck round bottom flask for 4 h in order to facilitate the de-bundling of the SWNT network. Meanwhile, an

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argon flushed 100-mL 2-neck round bottom flask equipped with a Drierite-filled drying tube and a condenser was charged with 0.2184 g naphthalene (1 eq.), 0.0111 g Li (1 eq.), and 10 mL of anhydrous THF. This mixture was then refluxed at 60 °C for 2 h. Naphthalene was added to serve as an electron carrier to allow the reduction of Mg(II) to Mg(0). The SWNT dispersion was then added via syringe into the dark green Li-naphthalide solution and was stirred at room temperature for 24 h. To the SWNT-Li-naphthalide dispersion, 0.264 mL of methylmagnesium chloride (MeMgCl) (0.5 eq.) was then added and the reaction was refluxed at 60 °C for 24 h. The solution was centrifuged under argon (using capped centrifuged tubes). The resulting solid SWNTs sediments were washed several times in anhydrous THF and were dried under vacuum at room temperature. The dried SWNTs-Mg nanoparticle composites were stored under argon until analysis.

Characterization of SWNTs-Mg NPs composites: HR-TEM analyses were carried out on a JEOL JEM 2100F, operated under 200 kV and equipped with EDS. The sample under analysis was sonicated in anhydrous THF and deposited onto copper grids coated with lacey carbon film (300 mesh). The EDS analyses were performed using a 0.2 nm spot size. Raman spectra were obtained on solid samples dispersed in ethanol and placed onto a Si wafer. Spectra were obtained on a Jobin-Yvon Horiba Labram Raman microspectrometer with excitation from an argon-ion (514.5 nm) laser. A CCD detector from Andor was used.

3. Results and discussion

Although we have explored several conditions for the preparation of SWNTs-Mg NP composites, our observations indicated that the most uniform distribution of Mg nanoparticles onto SWNTs, without additional chlorinated byproducts, was obtained when a ratio of 1:1:0.5 equivalents of lithium:naphthalene:MeMgCl respectively was used with 120 mg of SWNTs. Fig. 1a shows the HR-TEM image of the untreated starting SWNTs which were indeed very pure and relatively free from amorphous carbon and metal impurities. Bundles of SWNTs were observed ranging in size from 4 to 20 nm in diameter. The EDS data confirmed that the

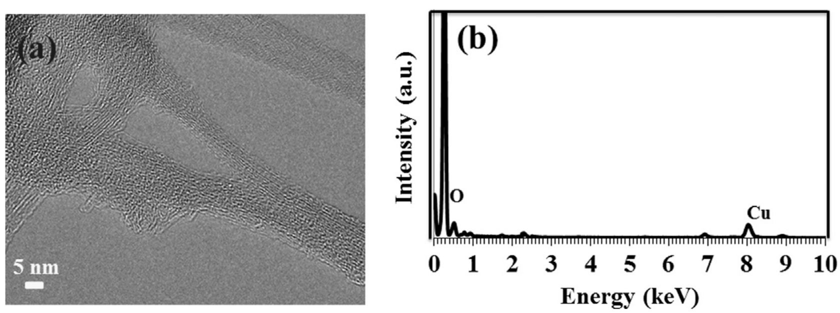

Fig. 1. (a) HR-TEM image of untreated SWNTs. (b) EDS spectrum of untreated SWNTs indicated the absence of magnesium. The Cu signal originated from the TEM sample grid.

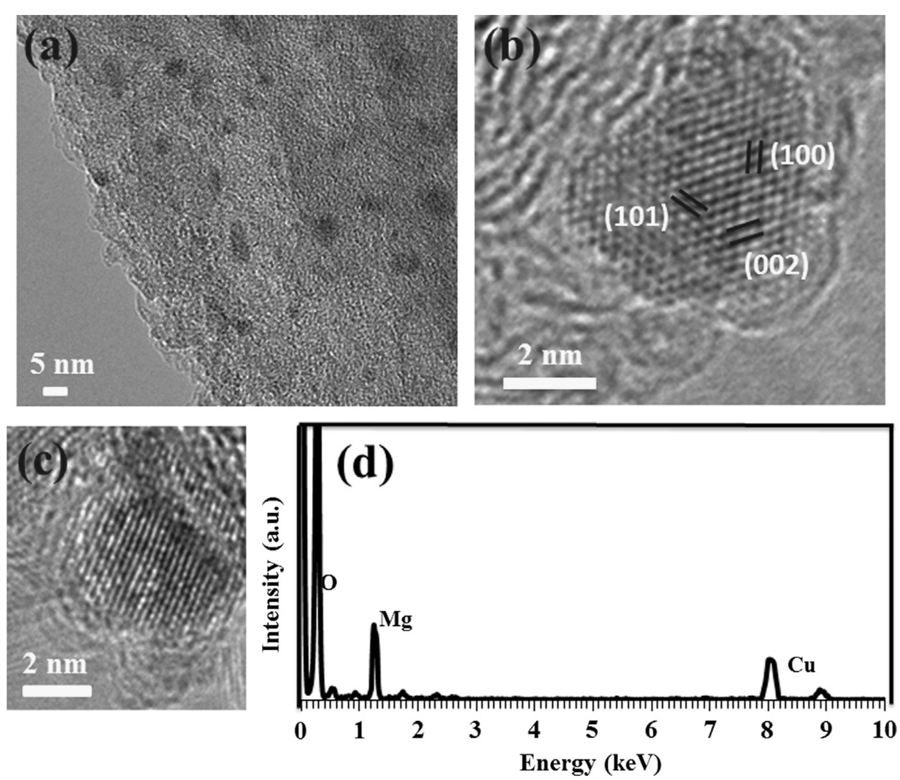

Fig. 2. (a)–(c) HR-TEM images of treated SWNTs. (a) Image shows a uniform distribution of Mg nanoparticles on the SWNTs with an average particle size of 4.5 ± 0.5 nm. (b) and (c) Images show lattice fringes of the hexagonal metallic magnesium nanoparticles (JCPDS card # 035-0821) (d) EDS spectrum of treated SWNTs indicated the presence of magnesium. The Cu signal originated from the TEM sample grid.

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