Exfoliated Molybdenum Disulfide as a Platform for Carbon Nanotube Growth—Properties and Characterization

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ABSTRACT: Carbon nanotubes (CNTs) have been of great interest because of their unique electrical, structural, and mechanical properties. Many methods for obtaining CNTs are known. Chemical vapor deposition (CVD) has been recognized as the most popular and practical synthetic method for obtaining CNTs, with high purity, high yield, and low cost. Catalyst components are usually transient metals such as Fe, Co, and Ni, and hydrocarbons are used as a feedstock for the CNT synthesis. The metal particles are supported on the inorganic porous materials, such as alumina (Al2O3), silica (SiO2), magnesia (MgO), zeolite, and mesoporous silica. In this work, we propose a new platform for the deposition of metal nanoparticles and the growth of CTs. Molybdenum disulfide (MoS2) has gained much attention in the material fields. The principal aim of the present work is to compare the synergetic effect of MoS2 and CTs and to investigate the possibility of using the material in various fields. The obtained material was tested for its use in fire retardation. We compared the effect of adding bulk MoS2 and MoS2/CTs into the polymer matrix.

INTRODUCTION

The discovery of carbon nanotubes (CNTs) in 1991 by Iijima has stimulated intensive research to characterize their structure and determine their properties both in fundamental science and in industrial applications.1−5 On the basis of CNTs as active components, many potential applications such as field emitters, nanoscale transistors, supercapacitors, and sensors have been demonstrated. There are various mechanisms of CNT growth. The popular mechanism includes four steps such as dissociation of hydrocarbons, migration of carbon, segregation of carbon, and formation of graphene sheets. The common characteristic of all mechanisms is to provide energy to a carbon source for the creation of carbon atoms that generate CNTs.6−10 There are several techniques that have been developed for producing CNTs: the chemical vapor deposition (CVD) technique, the laser ablation technique, and the carbon arc discharge technique.11−13 A CVD method has been widely used for the synthesis of nanotubes because carbon feedstock, such as hydrocarbon, can be fed continuously into the reaction.14 This is different from the other methods, which use carbon vapor evaporated from metal-filled graphite rods and thus supply a limited amount of amorphic carbon for nanotube growth. Moreover, the CVD growth can be performed at a lower temperature (600−1000 °C) compared with other methods, allowing nanotube growth.15−17

The structures of nanoscale carbons formed by the CVD method strongly depend on the kind of molecules because of their carbon sources as well as the type of metal catalysts. Various types of catalysts are used. These are used either in the form of an element or in the form of a salt, which is supported on different inert materials.18−20 Different monometallic, bimetallic, and trimetallic catalytic compounds with different supports, especially Fe/MgO, Ni/MgO, Mo/MgO, Co/MgO, Fe/Al2O3, FeNi/MgO, FeZn/MgO, FeCo/MgO, CoNi/MgO, Mo Ni/MgO, CoMo/MgO, CoNi/SiO2, FeCo/Al(OH)3, Fe CoMo/Al(OH)3, FeNiMo/Al(OH)3, and so forth, have been explored for the production of CNTs. They produce different types of CNTs ranging from single-walled nanotubes (SWNTs), double-walled nanotubes (DWNTs), and multi-walled nanotubes (MWNTs).21−25

Another very important aspect of CNT growth is the selection of a suitable substrate for the deposition of catalyst particles. Different materials such as magnesia (MgO), silica (SiO2), alumina (Al2O3) zeolite, and mesoporous materials such as MCM-41 have been studied.26−28 MgO support appears promising as it provides a high yield of surface-clean SWNTs and DWNTs and is easily removed by a simple acid treatment. Use of these materials as a support for catalysts has a lot of disadvantages. Coquay demonstrated that the MgO-supported catalysts are efficient for carbon nanotube (SWNT and DWNT) synthesis, and MgO can be removed by an acid solution such as hydrochloric acid. The disadvantage was that the CNTs prepared by MgO-supported catalysts often contained carbon impurities, for example, amorphic carbon,
that cannot be removed by an acid treatment. These impurities were caused by the existence of unwell dispersed metal oxide species on the MgO-supported catalysts.32 Zhao obtained CNTs that were grown on the surface and edge of graphene by CVD at 1000 °C. The isolated graphene lamellae were connected by CNTs to form a spatial network structure of CNTs that provides the effective electron transmission channels.33 Also, Dong obtained highly conductive graphene–CNT hybrid materials that were synthesized on a Si NP-coated copper foil by a one-step CVD method. This easy method provides a novel way to obtain nanocarbon hybrids.34

In this work, we present a new method by which CNTs and nanoparticles were obtained from the decomposition products of NiO, and Fe2O3 when the reaction was performed over MoS2. A detailed study on the effect of various metal oxides to growth of carbon tubes (CTs) upon the morphology of the formed products and their properties has been discussed. The synergistic effect of MoS2—support and CNTs was investigated. Flame retardancy of polymeric materials is conducted to provide fire protection to flammable consumer goods, as well as to mitigate fire growth in a wide range of fires. This paper shows new support for CNTs, which can be used in the commercial flame retardant technology. Scheme 1 shows the preparation of a MoS2 platform for the growth of CTs.

Scheme 1. Graphical Representation of the Experimental Steps To Obtain (1) Exfoliated MoS2, (2) Functionalized MoS2 Metal Oxide Nanoparticles, and (3) CTs Grown on MoS2 via CVD

### EXPERIMENTAL SECTION

**Materials.** 1-Methyl-2-pyrrolidone (anhydrous, 99.5%), bulk MoS2 (powder), nickel(II) acetate tetrahydrate (98%), iron(II) acetate (95%), and polyethylene (PE) (average Mw ≈ 4000 by GPC, average Mn ≈ 1700 by GPC) were purchased from Sigma-Aldrich.

**Exfoliation of MoS2.** The initial MoS2 concentration experiments were performed by adding 1 g of powder to 10 mL of N-methyl-2-pyrrolidone (NMP) in a 100 mL capacity, flat-bottomed beaker. The solution was sonicated continuously for 72 h using a horn probe sonic tip. The beaker was connected to a cooling system that allowed cold water (15 °C) to flow around the dispersion during sonication. The final dispersion was centrifuged four times at 1500 rpm.

**Preparation of MoS2/NiO and MoS2/Fe2O3.** Two samples of MoS2 modified by metal oxide nanoparticles (MoS2/NiO, and MoS2/Fe2O3) were prepared according to the following procedure: 150 mg of MoS2, and 150 mg of nickel (II) acetate tetrahydrate (product referred to as MoS2/NiO), and iron(II) acetate (product referred to as MoS2/Fe2O3) were dispersed in 250 mL of ethanol and sonicated for 2 h. Afterward, the mixture was stirred for another 24 h. Finally, the sample was dried in high vacuum at 440 °C for 3 h.

**Growth of CTs.** The simplest system to grow CTs by CVD consists of catalyst nanoparticles brought to high temperature in the presence of gaseous carbon-containing molecules. The nanoparticles are commonly made of a transition metal such as Fe or Ni. The catalyst particles are deposited in the last step on a support material. CVD processes were performed in a quartz tube placed inside a tube furnace. The process tubes were heated in air at 800 °C for 1 h prior to both CVD and annealing. Ar and C2H4 were used for CVD as a carrier gas and carbon source, respectively. In a typical synthesis, 1 g of the sample was first placed on a ceramic boat and inserted into the process tube. Samples were heated at 800 °C under a flow of 100 sccm of argon and at a given temperature with a flow of 60 sccm of C2H4. After 15 min, the ethylene was turned off and the system was cooled to room temperature.

**Preparation of Composites.** PE was used as a polymer matrix. The contents of MoS2/NiO, MoS2/Fe2O3, MoS2/CTs@NiO3, and MoS2/CTs@Fe2O3 are 1, 5, 3, 6, and 10 wt %, respectively. After being dried at 70 °C for 12 h, MoS2 samples were blended with PE using a two-screw extruder at a temperature of 120 °C.

**Characterization.** X-ray diffraction (XRD) was carried out on a Philips diffractometer using Cu Kα radiation. Transmission electron microscopy (TEM) and high-resolution TEM were performed on a Tecnai F30 transmission electron microscope (FEI Corporation, USA) at an acceleration voltage of 200 kV. Thermogravimetric analysis was carried out using a SDT Q6000 thermoanalyzer instrument (TA Instruments Inc.) under an air flow of 100 mL/min. In each case, the samples were heated from room temperature to 900 °C at a linear heating rate of 10 °C/min. The samples were measured in an alumina crucible with a mass of about 5.0 mg. Raman spectroscopy was performed using a microprobe mode using the following lengths of the laser: 785 nm (InVia Renishaw). Raman spectroscopy is an ideal method to study the structural properties of the nanomaterials. Atomic force microscopy (AFM NTegra Aura (NT-MTD) microscope) determined the number of layers in the MoS2 samples. The polymer composites will be prepared via a standard technique employing a laboratory twin-screw extruder (Zamak Mercator EHP 2x12). It is widely used in polymer research and can be used to prepare all kinds of polyolefin-flame-retardant nanocomposites. The measurements of the flame retardancy will be realized via a micro calorimeter (FAA MICRO Calorimeter).

### RESULTS AND DISCUSSION

The successful exfoliation of MoS2 with NMP was confirmed by TEM and AFM. The morphologies of MoS2 nanosheets before and after the sonication treatment were investigated by TEM. Figure 1A-'A' shows the TEM images of bulk MoS2 large area stacked nanosheets. After sonication (Figure 1B-B'), the seriously stacked nanosheets of bulk MoS2 were exfoliated to a thinner layered structure observed by a lighter contrast. The dimensions of the flakes were characterized by AFM imaging (Figure 1A-B). The AFM profiles exhibit a flat and uniform surface without aggregation, an indication that homogeneously two-dimensional flakes were obtained. It is noted that the exfoliated flakes produced with NMP were typically 1.5–3.5 nm in height, which corresponds to five to six layers of MoS2 (single layer thickness: ~0.7 nm).35,36

Exfoliation of the new material MoS2 into few layers largely preserves their properties and also leads to additional characteristics because of confinement effects. The MoS2 exfoliation experiments were performed by adding the powder to NMP in a flat-bottomed flask. These mixtures were sonicated continuously for 72 h using a horn probe sonication...
tip. The flask was connected to a cooling system that will allow cold water (15 °C) to flow around the dispersion during sonication. After sonication, the suspensions were centrifuged with a defined speed to control the number of layers in the final MoS$_2$ sample. The solvent was filtered from the sample before the analysis. Raman spectroscopy (Figure 2) has been used to determine the number of layers, as well as to examine the changes in material properties with thickness. This is commonly accompanied by a change in the intensity of these peaks, which has also been observed for the investigated samples of this study. The Raman spectra were excited by a 785 nm line in an air ambient environment. $E_{2g}^1$ ($\sim 380$ cm$^{-1}$ for bulk MoS$_2$) and $A_{1g}$ ($\sim 406$ cm$^{-1}$ for bulk MoS$_2$) modes are observed in both exfoliated and bulk MoS$_2$. It is proved that the frequency of the $E_{2g}^1$ peak decreases while that of the $A_{1g}$ peak increases with an increasing layer number. When the number of layers changes, the interlayer van der Waals force in MoS$_2$ suppresses the atom vibration, resulting in higher force constants. $^{34,35}$

In the next step, the samples of MoS$_2$ were modified by metal oxide nanoparticles (Ni$_2$O$_3$ and Fe$_2$O$_3$). The obtained samples were investigated using TEM. Figure 3 shows typical TEM images of the metal oxide nanoparticles supported on the MoS$_2$ surface. The nanoparticles are highly dispersed on the MoS$_2$ surface and deposited homogeneously. The detailed TEM analysis of the Ni$_2$O$_3$ samples allowed us to reveal the diameter distribution of the samples. It is noted that the diameter of Fe$_2$O$_3$ nanoparticles (Figure 3A,A') are centered at 2–5 nm and that of Ni$_2$O$_3$ nanoparticles (Figure 3B,B') are centered at 10–13 nm.

To confirm the crystal-phase composition of metal nanoparticles supported on MoS$_2$, XRD analysis was used. Figure 4A,B shows representative data for MoS$_2$/Fe$_2$O$_3$ and MoS$_2$/Ni$_2$O$_3$, respectively. All sharp diffraction peaks (red cubes) were detected at $2\theta \cong 14, 33, 40, 44^\circ$ corresponding to MoS$_2$. A few strong XRD peaks in Figure 4A (blue cubes) are attributed to Fe$_2$O$_3$. All these diffraction peaks are in good agreement with that of the standard pattern for rhombohedral $\alpha$-Fe$_2$O$_3$. $^{36}$ The second major peak shown in Figure 1B at $2\theta \cong 50, 56^\circ$ is typical for Ni$_2$O$_3$. The characteristic peaks for Ni $2\theta \cong 58, 73, 76^\circ$ and NiO$_2$ $2\theta \cong 36, 61^\circ$ are observed.

The as-prepared MoS$_2$ substrates with metal oxides were used in the CVD process for CT growth. The morphology of the samples was also determined by TEM (Figure 5). The
TEM images of MoS$_2$/Fe$_2$O$_3$ and MoS$_2$/Ni$_2$O$_3$ after CVD clearly show tubular structures, indicating the activity of the catalysts. The analysis of the images allows us to calculate the diameter distribution of the tubes in both samples. We studied that the size of the original iron and nickel oxide nanoparticles become bigger during CVD, which resulted in the growth of CNTs with larger diameter when reacted with ethylene. Additionally, the samples do not contain undesired carbon impurities such as amorphous carbon or graphitic nanoparticles, which are typically present when other supports are applied.
in the particles size is clearly due to the weak interaction between the catalysts and the MoS₂ support (Figure 6A,B). It reflects strongly limited thermal diffusion of metal atoms into the support matrix. This observation is opposite to the interaction between the catalysts and MgO. Here, the strong metal—support interaction yields in a smaller diameter of the catalysts and a smaller mean diameter of the grown tubes. It was found that the original iron particles became smaller during CVD and that these small particles resulted in SWNTs and DWNTs when reacted with methane.²³ In these cases, we proposed the root growth mechanism. When the catalyst—support interaction is strong, hydrocarbon decomposes on the top surface of the metal, carbon diffuses down through the metal, and the CT precipitation fails to push the metal particle upward. Next, hydrocarbon decomposition takes place on the lower peripheral surface of the metal, and the as-dissolved carbon diffuses upward. Thus, CTs grow up with the catalyst rooted on the support.³⁸

Also, the CNTs prepared by MgO—support catalysts often contain carbon impurities, such as carbon nanofibers, amorphous carbon that cannot be easily removed by an acid treatment. These disadvantages are caused by the existence of unwell dispersed metal oxide on the MgO—support catalysts. This is not a case when MoS₂ is applied.³⁶,³⁷

**CONCLUSIONS**

The catalytic performance of NiₓOᵧ, FeₓOᵧ catalysts supported on MoS₂ for the formation of CNTs through ethylene decomposition was investigated. A weak metal—support interaction has been revealed, which led to an increase in the diameter of CTs. Interestingly, the samples do not contain unwanted carbon impurities such as amorphous carbon or graphitic nanoparticles, which are typically present when other supports are applied. Mutual synergy between MoS₂ catalyst, and CTs resulted in better thermal response compared to PE/MoS₂ composites. Better values of pHRR, THR, and HR capacity were obtained for the samples of MoS₂/CTs@FeₓOᵧ and MoS₂/CTs@NiₓOᵧ compared to pristine polymer, polymer with MoS₂, or with MoS₂ with metal particles. Therefore, we prove that the as-produced samples are useful for certain applications and no additional steps, including purification, are required prior utilization.

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**Notes**

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