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Preparation of Multiwall Carbon Nanotubes/Alumina Precursor Composites†

JING WANG*, YONG XU and MINGXU ZHANG

Institute of Material Science & Engineering, Anhui University of Science & Technology, Huainan 232001, P.R. China

*Corresponding author: E-mail: jinwang@aust.edu.cn

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In this study, the multi-walled carbon nanotubes (MWCNTs) were modified with a mixture of concentrated HNO_3/H_2SO_4 (3:1 by volume). The TEM showed that the modified MWCNTs were well dispersed. Compared with raw MWCNTs, the modified MWCNTs can disperse uniformly in a mixture of saturated aqueous solution of NH_4HCO_3, $NH_3{\cdot}H_2O$ and little sodium dodecyl benzene sulfonate (SDBS) solution. The TEM and XRD of the powders of composites indicated that the alumina precursor may wrap the MWCNTs absolutely.

Key Words: Multi-walled carbon nanotubes, Modified, Concentrated HNO_3/H_2SO_4, Alumina precursor.

INTRODUCTION

Carbon nanotubes (CNTs) have been regarded as ideal reinforcements of high-performance composites with enormous applications because of their novel structure and remarkable mechanical, thermal and electrical properties[1]. However, they have a tendency to aggregate together and it is difficult to disperse them because of CNTs show high, long-range van der Waals forces of attraction. Therefore, dispersion and functionalization of the CNTs have received much attention recently.

A mixture of concentrated H_2SO_4/HNO_3 (3:1 by volume) can be used to modify CNTs and more effective than nitric acid in removing impurities[2,3]. The modified CNTs can be widely used in composites field and most researchers focus attention on reinforced CNTs/polymer composites and CNTs/metal composites[4,5]. In this study, we used *in situ* coating process to prepare multi-walled carbon nanotubes (MWCNTs)/alumina precursor composites.

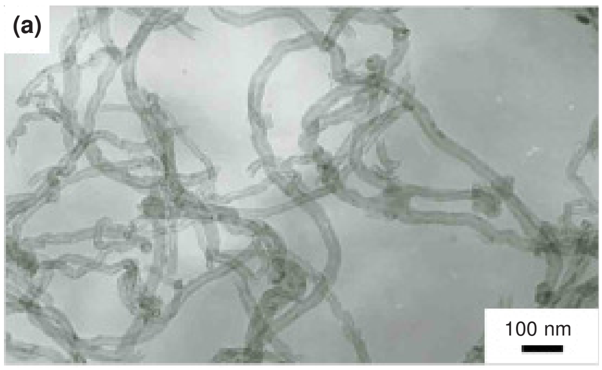
EXPERIMENTAL

Surface modification method: Multi-walled carbon nanotubes (MWCNTs) (1 g) were taken with 80 mL a mixture of concentrated HNO_3/H_2SO_4 (3:1 by volume) in a 100 mL round-bottomed flask. The mixture was sonicated for 10 min in an ultrasonic bath at room temperature. The resulting mixture was stirred magnetically at 80 ºC in oil bath for 50 min.

Preparation of MWNTs/alumina precursor composites: 8.885 g $NH_4Al(SO_4)_2{\cdot}12H_2O$ and 8.674 g NH_4HCO_3 were dissolved into deionized water to make saturated aqueous solution at room temperature. $NH_3{\cdot}H_2O$ was used to keep the pH in the reaction process and the sodium dodecyl benzene sulfonate (SDBS) was dissolved into the mixture of $NH_3{\cdot}H_2O$ and NH_4HCO_3. After that, 0.003 g acid treated MWCNTs were added into the mixture to form a black solution with the help of ultrasonic bath for 20 min. After that, the saturated aqueous solution of $NH_4Al(SO_4)_2{\cdot}12H_2O$ was added dropwise to the mixture.

RESULTS AND DISCUSSION

Comparison of dispersion analysis indicated that the modified MWCNTs were dispersed well, the raw MWCNTs were gathering together and the length of the modified MWCNTs was similar to the raw MWCNTs (Fig. 1).



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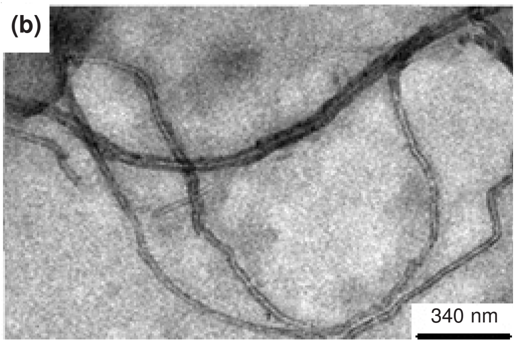

Fig. 1. TEM image of raw (a) and modified (b) MWCNTs

Comparison of dispersion analysis indicate that the suspension stability of raw MWCNTs is very poor as seen in photographs since it easily get settle down at the bottom (Fig. 2). This is due to the agglomeration of MWCNTs. Moreover, the modified MWCNTs showed better suspension stability. This is due to the presence of -OH and -COOH functional groups on the surface of the modified MWCNTs, which result in the repulsion of MWCNTs from each other and keep the solution dispersed.

Fig. 2. Visual confirmation of suspension stability of modified MWCNTs in a mixture of saturated aqueous solution of NH_4HCO_3, $NH_3{\cdot}H_2O$ and little sodium dodecyl benzene sulfonate (SDBS) solution. (a) raw MWCNTs (b) modified MWCNTs

Fig. 3 shows that the alumina precursor may wrap the MWCNTS absolutely and few modified MWCNTs dispersed well in the alumina precursor. The needlelike structure powders were $NH_4Al(OH)_2CO_3$, which could confirmed by the XRD of composites (Fig. 4). The needle like structure powders were getting together, which may due to the high concentration of the $NH_4Al(SO_4)_2{\cdot}12H_2O$ solution.

Fig. 4 indicates that the product is a mixture of two phases $NH_4Al(OH)_2CO_3$ and MWCNTs. Well-resolved diffraction peaks reveal the good crystallinity of the $NH_4Al(OH)_2CO_3$ specimens. The diffraction peak at $2\theta = 26.2°$ is assigned to (002) plane of MWCNTs. No peaks corresponding to impurities are detected. So it could be concluded that a kind of MWCNT/$NH_4Al(OH)_2CO_3$ composites was formed during the reactions.

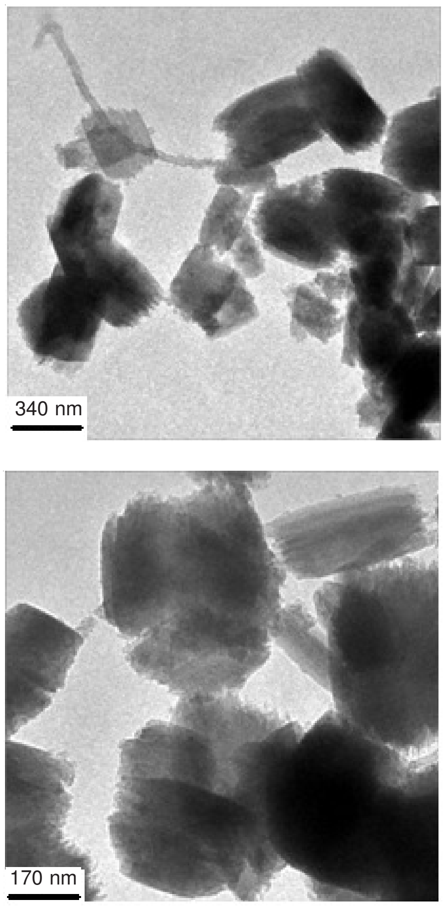

Fig. 3. TEM images of MWCNTs/alumina precursor composites

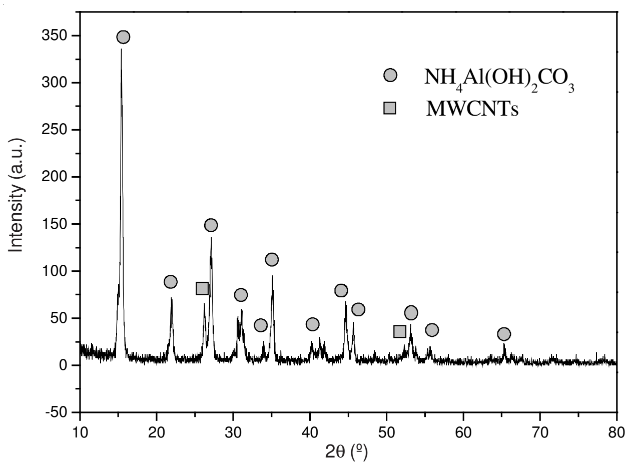

Fig. 4. XRD pattern of the MWCNTs/alumina precursor composites

Conclusion

In summary, we developed a simple, effective and reproducible method for preparing well dispersed MWCNTs and MWCNTs/$NH_4Al(OH)_2CO_3$ inorganic hybrid material. The modified MWCNTs can disperse uniformly in a mixture of saturated aqueous solution of NH_4HCO_3, $NH_3{\cdot}H_2O$ and little sodium dodecyl benzene sulfonate solution. Further work should be done on investigating not only the dispersion properties of MWCNTs but also the relation between the $NH_4Al(SO_4)_2{\cdot}12H_2O$ concentration and the coating property of $NH_4Al(OH)_2CO_3$.

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