

Molybdenum Disulfide Sheathed Carbon Nanotubes

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Abstract: Single and double layered MoS₂-coated multiwalled carbon nanotubes (MWCNs) were successfully prepared by pyrolyzing (NH₄)₂MoS₄-coated multiwalled carbon nanotubes in an H₂ atmosphere at 900 °C. MoS₂-coated MWCNs would be expected to have different tribological and mechanical properties compared to MoS₂, so it may have potential applications in many fields.

Keywords: Carbon nanotubes, MoS₂, coating, inorganic fullerene-like.

Inorganic compounds with 2D layered structures are unstable against bending and form hollow, closed clusters designated as inorganic fullerene-like (*IF*) structures. The analogy between graphite and 2D layered compounds was pointed out initially by Tenne and co-workers^{1,2} in the case of MoS₂, WS₂, and the respective selenides.

The folding of triple-layered MX₂ (M=Mo, W; X=S, Se) lattices into closed structures is, however, more complicated than in the case of single sheet graphitic structures. The triple S-M-S layer is unable to form odd-membered rings (important in graphite curvature) because the W/Mo atoms can only bond to S³. So it is difficult to form single-walled MX₂ nanotubes. But, if there is a template, which is possible to obtain single-walled MX₂ coated nanotubes. Recently, CNTs have been used as templates to grow MoS₂, WS₂ and NbS₂ coated carbon nanotubes, some of them contain 1-2 layers of the chalcogenide at the exterior⁴⁻⁸. The CNTs were coated with the metal oxide and treated in an H₂S/H₂/N₂ atmosphere at elevated temperatures to convert the oxide to sulfide. However, the CNT core was not removed in the nanostructure.

Herein we report the successful production of single- and double-layered MoS₂-coated multiwalled carbon nanotubes (MWCNs) by treating (NH₄)₂MoS₄ coated MWCNs in H₂. The formation of MoS₂-coated MWCNs implies that (NH₄)₂MoS₄ is converted into MoS₂ directly on the MWCN surfaces. Toward this goal is to deposited (NH₄)₂MoS₄ onto MWCNs first. The wetting of MWCNs is difficult, due to high surface tension⁹. Preliminary attempts involved oxidation of MWCNs *prior* to the oxide coating process. However, this method causes significant damage to the MWCN surface and lowering the yield of coated materials. We use (NH₄)₂MoS₄ as starting material, which can be dissolved in water, at the same time easily adsorbed onto the

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surface of MWCNs. In the present work the damaging of the surface of MWCNs has been avoided. This is the difference from that reported in the references.

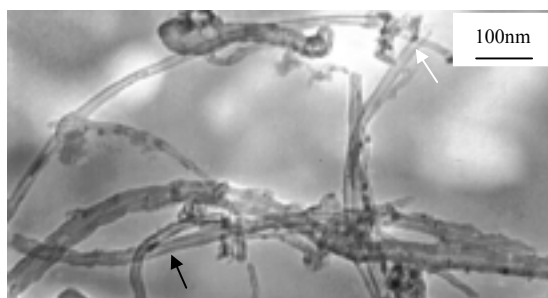
Experimental

MWCNs (50 mg), produced by catalytic chemical vapor deposition over Co-Mg-O catalyst, were sonicated in alcohol for 1 h. To this suspension was added 3 mL of 1%(weight percent) aqueous solution of $(\text{NH}_4)_2\text{MoS}_4$. The mixture was kept on sonicating for another hour and dried at 60 °C. Finally, the solid residue was heated at 900 °C, in a hydrogen atmosphere for 45 min. The resulting black product was subjected to TEM, HRTEM, EDX and XRD analysis. The reaction involved in the formation of the disulfide is as follows:



Results and Discussion

Figure 1 Overview of the obtained sample



According to TEM and HRTEM, 65% of the MWCNs were either fully or partially coated with dark material, in 1~3 layers. **Figure 1** (TEM image) is an overview of the obtained sample. We can see that the MWCN was (dark arrow) and was not (white arrow) sheathed with MoS_2 . **Figure 2** shows that MWCNs were mainly coated with single-layer of MoS_2 . There are also double and triple layer of MoS_2 . Image 2(b) was the partial magnification of image 2(a). From image 2(b), we can apparently see the carbon nanotube layers (white arrows) and the MoS_2 layer (dark arrow). EDX analysis was carried out on both partially and fully coated MWCNs. Only a carbon signal was present when the EDX probe was focused on the uncoated section of a partially coated MWCN (**Figure 3a**). When the probe was focused on the coated area, carbon, molybdenum and sulfur were detected (**Figure 3b**). Carbon, molybdenum and sulfur were always presented when the EDX probe was focused on a fully coated MWCN. The atomic ratio of molybdenum to sulfur was about 1:2(that is, MoS_2). It is clear from HRTEM and EDX that the coating material is MoS_2 and that the carbon signal aroused from the MWCNs. The XRD pattern of the MoS_2 -coated MWCN sample is shown in **Figure 4**. The MWCN reflections mostly overlap with those of MoS_2 . The MoS_2 reflection intensity is lower than that displayed by the MWCNs. The only reflection arising from the coated-MWCNs corresponds to the (002) line at *ca.* $2\theta=26.5^\circ\text{C}$, similar

to that of uncoated-MWCNs. The XRD pattern reveals that MoS₂ has a well-developed crystalline structure.

Figure 2 TEM images of a MWCN coated with single, double and triple layer of MoS₂. Image (b) was the partial magnification of image(a)

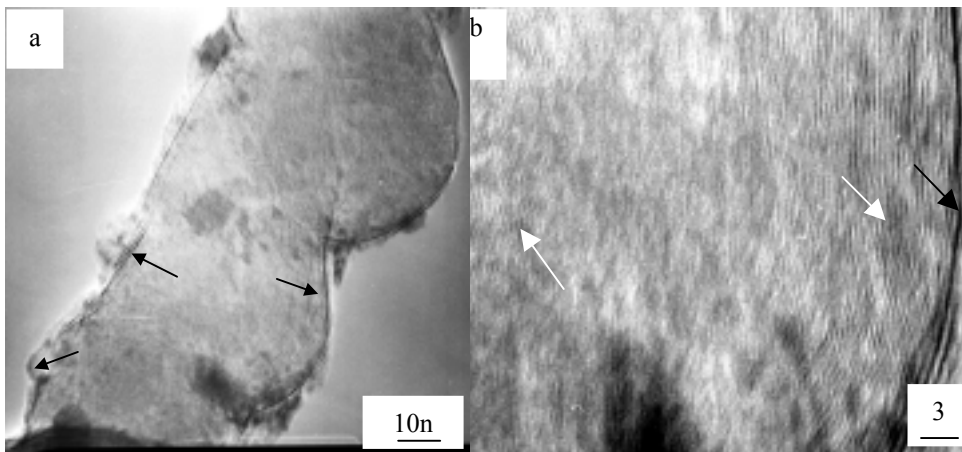


Figure 3 EDX patterns of uncoated MWCNs (a) and MoS₂-coated MWCN (b) (The copper signal arises from the TEM grid)

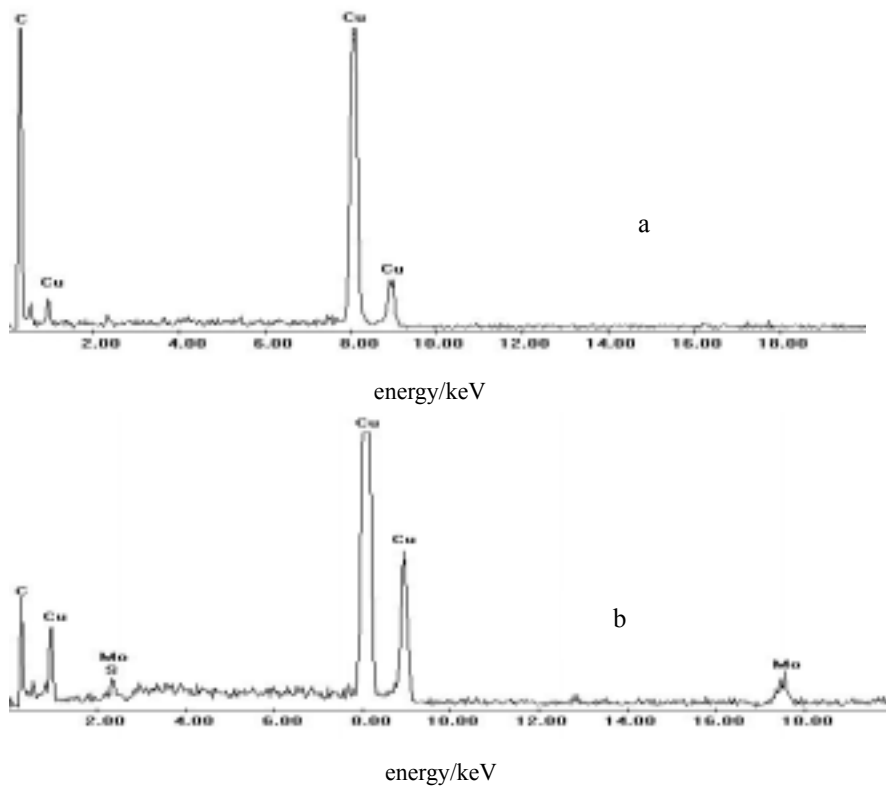
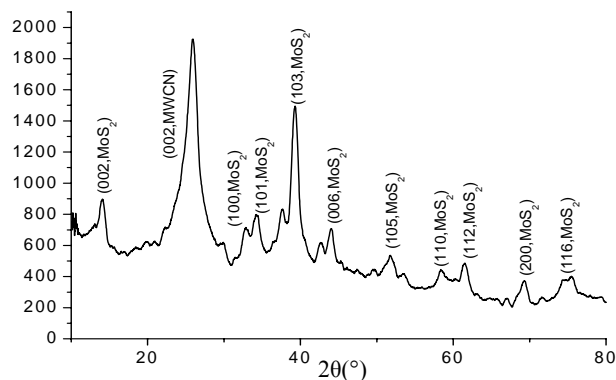


Figure 4 XRD profile of a sample containing MoS₂-coated MWCNs

Conclusion

In conclusion, we have successfully fabricated single and double layered MoS₂-coated multiwalled carbon nanotubes (MWCNs) by a very simple method. Due to its composite nature, MoS₂-coated MWCNs would be expected to have different tribological and other properties compared to MoS₂. Further work on optimizing their formation conditions and improve their yields is in progress.

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