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Title of Ph. D. Thesis : "Purification of as grown carbon nanotubes (CNTs) and its
characterization by SEM, AFM and Raman Studies"

Abstract

In this work, we firstly study the problem of purification of as-produced carbon nanotubes (CNTs) material. We have investigated the effects of chemical treatment on the Single Wall Nanotubes (SWNTs) before and after being modified with sulfuric acid and nitric acids (H_2SO_4/HNO_3) by FESEM, Raman, FTIR and UV-Vis-NIR spectroscopy. The FTIR results show successful carboxylation of the CNT sidewalls. We observe some important Raman features: Radial breathing mode (RBM), Tangential mode (G-band), and Disordered mode (D-band); which are affected due to the chemical oxidation of carbon nanotubes. A relative increase in the intensity ratio of the disordered peak, D-band, confirmed the functionalization. This increase was attributed to a change in the CNTs structure due to the introduction of the –COOH and –OH groups onto the walls of the nanotubes. This successful functionalization is achieved in 6-8 hrs of refluxing. We have found that the ratio of D- and G-band intensity (I_D/I_G), increase after acid treatment and the RBM mode in acid treated SWNTs is almost disappeared. This is the indicator of the level of the purification and functionalization of the CNTs.

Purification of Multiwall carbon nanotubes (MWNTs) is also investigated to determine the structural and chemical changes in atomic bond. As-grown MWNTs were treated in acid

mixture under a refluxing condenser with magnetic stir for different time period 5hr, 7hr, 9hr, and 15hr at 70 °C in order to remove amorphous carbon and catalysts completely. The results of Raman spectroscopy suggest that the appropriate mixing ratio between H₂SO₄ and HNO₃ is 3:1 with treatment time from 5- 9 hr is suitable for purification and create fewer defects during acid treatment. FESEM images are clean after purification; this means we purified CNTs successfully.

Another method is also applied here for the purification of CNT. This method cause fewer defects on the CNT surface. UV irradiation was conducted under ambient condition in a UV lamp chamber which had a layer of reflective aluminium foil inside to ensure the entire sample would have an even exposure to UV light. The narrowing of D-band is consistent with highly purified nanotubes which are seen in our results. Raman result shows that the ozone oxidation can remove the defect site of nanotubes. Intensity ratio I_{D^*}/I_D increases continually with increasing the ozone oxidation time which confirm the reduction of impurities from the MWNTs. FESEM results confirms the removal of impurities.