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Synthesis of Transparent Carbon Nanotubes by Atomization of Ethanol in an Atmospheric Chemical Vapor Deposition System

Rontgen Gapusan[a*], Aian Ontoria[a], Wilbur Galarion[a], Ian Jasper Agulo[a], Alipio Garcia[a] and Chelo Pascua[a]

[a] University of the Philippines - Baguio

[*]E-mail: rontgen_009@yahoo.com

ABSTRACT

In this paper, we report on the chemical vapor deposition (CVD) synthesis of high-purity CNT using ethanol and ferrocene as carbon source and catalyst, respectively. An effective purification method was performed to increase the purity of the CNT films. In our experiment, the CVD system consists of a quartz tube with 24 mm inner diameter as the reaction chamber and a compressor nebulizer as the injection system. In a typical experiment, ferrocene is dissolved in 99.9% ethanol at 1 g/ 600 ml concentration. When the reaction temperature reached 1000°C, the solution was then injected into the reaction chamber using compressor nebulizer with argon as the input gas. Argon was also used as the carrier gas. The flow rate of argon gas was set to 2 L/min. After the synthesis, the reaction chamber was cooled down to room temperature. A transparent thin film was collected from the walls of quartz tube. SEM analysis reveals that the films consist of fiber-like structures, with no observable amorphous carbon and metal particles. Raman spectroscopy confirms the existence of carbon nanotubes. The CNT samples have narrow and high G-band (~1590 cm-1) and weak D-band (~1350 cm-1) intensities. The ratio of the intensities of D- and G- bands (ID/IG) provides the defect level within the graphene domain of the CNT. Raman Spectrum shows that relatively high quality CNT samples were synthesized due to their low ID/IG value.

The as-synthesized CNT were purified using three methods – i) ultrasonication with ethanol as the dispersive medium; ii) nitric acid (HNO_3) purification at 110ºC; and iii) peroxide and acid treatment. Ultrasonication of CNT films allows for the dispersion of CNT bundles from amorphous and other non-CNT nanostructures intertwined with the film. HNO_3 treatment is used to remove organic compounds and metallic catalysts from the CNT film samples. CNT undergoes peroxide treatment using 30% hydrogen peroxide (H_2O_2) for 24 hours, and the carbon nanotube film undergoes subsequent acid treatment using 37% hydrochloric acid (HCl) for another 24 hours. Raman spectra of the purified samples reveal that a significant decrease in the value of ID/IG was achieved using peroxide and acid treatment.

KEYWORDS: carbon nanotubes; chemical vapour deposition; peroxide treatment; acid treatment