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Assessment of the Reaction Time on the Morphology and Quality of Carbon Nanotubes – Silica Microparticles

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Abstract

Carbon nanotube has been grafted in-situ on the surface of spherical silica gel via floating-catalyst chemical vapour deposition method. The reaction temperature was set to be 760°C and 5 wt. % of ferrocene catalyst (dissolved in toluene) injected into the furnace at a rate of 0.04 ml/min. The reaction time was varied from 1 hour to 8 hours, with one hour interval. It was found that the reaction time of 3 hours yields the best quality hybrid particles. Prolonging the reaction time more than 3 hours resulted in the formation of CNT that consists of thicker tubes, based on the observation via Field Emission Scanning Electron Microscope (FESEM) and Transmission Electron Microscope (TEM). Secondary overgrowth was observed via TEM for tubes synthesized at 7 hours and 8 hours. These results were in agreement with Raman Spectroscopy analysis where the I_G/I_D ratio were very small, indicating high defects and impurities in the samples synthesized at reaction time higher than 3 hours.

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1. Introduction

Carbon Nanotube is known as a multifunctional filler that may impart an exrtraordinary mechanical properties[1], electrical properties[2], and thermal properties[3] in a composite. One big challenge faced during the composite processing is CNT agglomeration due to strong Van der Waals interaction and physical entanglements originating from their growth process. It has been reported elsewhere that direct growth of CNT onto fibres[4-6] and

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microparticles[7-9] could address issues related to dispersion. The CNT growth on silica[5], stainless steel[10], and alumina particles (μAl_2O_3) [9] are also described where an increase in thermal conductivity in an epoxy matrix is achieved [11].These substrates not only function as the growth sites, but also as transport medium to carry CNTs within the matrices.

Prolonging the reaction time results in supplying more carbon and catalyst source. For example, the CNTs yield on silica fibre increased to 4.93 mg/cm^2 from 0.09 mg/cm^2 by increasing reaction time from 15 to 240 minutes [5]. The growth of CNT on silica microparticles have been described in previous publication, where the growth time up to 3 hours has already been investigated [12]. This paper aims to investigate further on the effects of increasing the reaction time up to 8 hours, on the morphology and quality of the CNT.

Nomenclature

CNT	Carbon Nanotube
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
ID	Inner Diameter
OD	Outer Diameter

2. Experimental

The CNTs were grafted on spherical silica gels with pore size of 6 - 8 nm at 760°C, as described in details in [12]. Prior to that, the ferrocene (5 wt. %) was first dissolved in toluene and preheated to 200°C before injected into the furnace at a flow rate 0.04 ml/min. The reaction time is varied from 1 to 8 hours, with 1 hour interval. The other parameters were kept constant such as reaction temperature, ferrocene concentration, and injection rate, while varying the reaction time.

The morphology of the particles was assessed via Philips XL 30 FEG SEM at 10 kV and Philips CM200 TEM. The pin stub with adhesive carbon disc was slightly pressed onto nanoparticles before having it coated with gold using Edward S150B Sputter Coater. The topography was then viewed at 10kV acceleration voltage. For TEM characterisation, the images of the particles were recorded and viewed under bright field mode. Raman analysis were performed to assess the quality of the synthesized CNT. The measurements were performed at least three times per sample to ensure data accuracy.

3. Results and Discussion

3.1 Morphology

Figure 1 shows the typical SEM images of the particles synthesized at 1 hour, 3 hours, 6 hours, and 8 hours. It can be deduced from high magnification images (Figure 1, left column) that prolonging the reaction time resulting in higher coverage of CNT on the silica surface. Full silica coating is observed for the samples synthesized at 3 hours and more (Figure 1d, 1f, and 1h). The CNTs remain entangled although the reaction time has increased further. The tubes appear thinnest when reaction time is at 3 hours. Further increasing reaction time results in the presence of thicker tubes. It is also observed that there are two types of tubes for the sample synthesized at 8 hours. The CNTs at the ‘bottom’ layer seem thinner compared to the CNTs at the ‘top’ layer (Figure 1g). It may be inferred that after 6 hours, additional carbon source contributes to the tubes thickening that protrude up to the top layer. The tubes at the bottom layer appear thinner as these thick CNTs at the top layer prevent the diffusion of carbon source to the tubes underneath.