UNIVERSITI TEKNOLOGI MARA

THE DOCTORAL RESEARCH ABSTRACT

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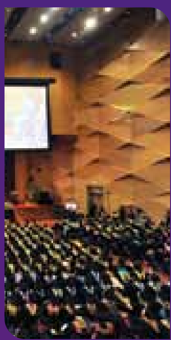

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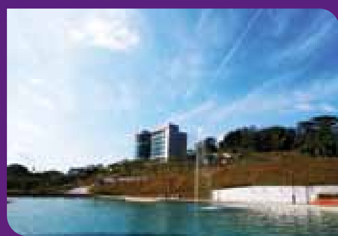

FIRST ISSUE

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Title : Carbon Nanotubes Growth From Camphoric Carbon Sources Using Transition Metal Catalysts
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This thesis covers the analysis of catalytic growth of carbon nanotubes (CNTs) under well-defined conditions, the optimization of the catalyst and introduces model for the growth mechanism based on the experimental results. Experimental investigations are presented to obtain a comprehensive picture on the catalytic growth of CNTs. The overall aim of this thesis is to deposit CNTs by the seeded catalyst method and the modified fluidized floating catalyst method by Chemical Vapour Deposition (CVD) and to investigate the effects of starting material and catalysts on the morphology and structure of the deposited CNTs. Camphor ($C_{10}H_{16}O$, crystalline state) and camphor oil (liquid state) are the precursor materials used as the source of CNTs. Transition metal (Fe, Ni, Co, Mn, Al, Mg) catalysts were prepared and the effect on their catalytic behavior were studied. Metal catalysts have been prepared by sol-gel method with or without support catalyst. Correlation between the catalyst particle size and CNT diameter has been the motivation to reduce the catalyst particle size down to nanoparticle size. Evolution of the catalyst particle size distribution during CNT synthesis is inevitable due to collision and evaporation of metal particles at high temperatures. In CVD the physical and chemical interactions between catalyst particle and support surface groups may be utilized in controlling the evolution of the particle size distribution. Physico-chemical properties of nanoparticles differ from the bulk values due to the high ratio of surface atoms to internal atoms. With varying catalyst loading with/without the catalyst support, the catalyst can form alloy and prevent the nanoparticles from segregating at high temperature. However, high fraction of the catalyst will form an isolated catalytic phase in the reduced catalyst. By controlling the mixture of catalyst can affect active catalyst particle, and also on the diameter of the CNTs. Catalytic carbon deposition reactions using camphor and camphor oil as the carbon containing feedstock have also been investigated over different type of transition metal catalyst with or without addition of support catalyst using seeded catalyst method and floating catalyst method. The CVD temperatures are varied between 650°C, 750°C and 850°C. Samples were characterized using a number of complementary techniques including, field emission scanning electron microscope (FESEM), Fourier Transmission Infrared (FTIR), X-ray diffraction (XRD) and Raman Spectroscopy. The findings from these techniques were used to explain the observed type and amount of carbon deposited. The subsequent studies of the morphology of the carbon structures grown by CVD revealed a significant influence of the deposition temperature and the catalyst material on the quality of the carbon structures. Fe/Ni/Mn was

found to be the most active catalyst to deposit CNTs by using camphor while Fe/Co/Al with ratio (1:2:1) was found to be the most active catalyst for camphor oil using seeded catalyst method with the deposition temperature of 850°C. However, high quality CNTs have been produced by fluidized floating catalyst method in which the precursor (camphor oil) and the catalyst Fe/Ni/Mg are placed in the same boat at moderate temperature of 650°C. The activity of metal catalyst was found to be dependent on a number of factors; mass of the catalyst, deposition temperature and type of precursor. It was found, in all cases that increasing deposition temperature resulted in higher deposition rates. Based on those experimental results a mechanism for the growth of CNTs is suggested. Carbon precursor dissociated catalytically on the catalyst nanoparticles spread on the catalyst boat. In the first stage, the carbon material reduces the metal oxide nanoparticles to pure metal. The further catalytic dissociation of carbon material presumably takes place at facets of well-defined crystallographic orientation and the carbon diffuses into the particle. The resulting density gradient of carbon dissolved in the particle drives the diffusion of carbon through the particle. In order to avoid dangling bonds, the carbon atoms assemble at a less reactive facet of the particle, which leads to the formation of a nanotube. Thicker nanotubes at higher temperatures are generated due to the dissociation of carbon material in the gas phase, which leads to the formation of carbon molecules that condense on the catalytically grown structures. Strong catalyst interaction between the catalysts is thought to be the dominant factor in improving the nanotube growth. In addition, the formation of a catalyst alloy is also possible in enhancing reactivity of the catalyst for the optimum growth of CNTs.