Carbon Nanotube Production over MCM-41 Type Catalytic Materials via CVD Method

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Abstract— We investigate the effects of mesoporous catalyst synthesis methods over carbon nanotube (CNT) production. Metal incorporated mesoporous catalysts were synthesized by two different microwave assisted synthesis methods. In the first method, MCM-41 mesoporous materials were synthesized with microwave radiation and then metal was impregnated into these as-synthesized MCM-41 samples. In the second method metal was added into the raw materials directly and then the mixture was treated in the microwave oven. The catalyst were tested in CVD for CNT production.

Keywords: MCM-41, CNT, CVD

Introduction

MCM-41 type catalytic materials with their hexagonal arrangement of one-dimensional pores and large surface areas (greater than 1000 m^2g^{-1}) attracted major attention of catalysis researchers in recent years [1]. Because of the unique structural features of the MCM-41, it is possible to produce narrow diameter distribution of carbon nanotubes (CNT) on this type of catalytic materials. Metal incorporated MCM-41 catalysts were synthesized by microwave-induced heating, which is a promising method due to several advantageous over conventional hydrothermal method. Due to the more uniform heating throughout the reaction vessel, the microwave-induced heating results in more homogeneous nucleation and shorter crystallization times significantly compared to the hydrothermal method [2].

Experimental

In this study, two different synthesis methods were investigated in catalyst production. In the first method, MCM-41 mesoporous materials were synthesized with microwave radiation and then metal was impregnated into these as-synthesized MCM-41 mesoporous materials using different Si/metal ratios as 25, 50, 75 and 100. In the second method metal was added into the raw materials directly and then the mixture was treated in the microwave oven using different Si/metal ratios as 25, 50, 75 and 100. The products obtained were characterized by XRD, N_2 physisorption, SEM, EDS and TEM.

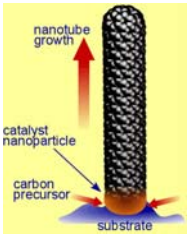

Figure 1. Schematic representation of CNT growth on the metal incorporated MCM-41 meseporous molecular substrate

Chemical Vapor Deposition (CVD) is a good method of conversion of acetylene gas to carbon fibers or carbon nanotubes by using metal catalysts. It is important that metal dispersion on the catalyst as well as metal particle size is important on the growth of CNTs.[3] Alignment may be improved with better control of the density of catalytic sites by means of a template of appropriate pore structure. Mesoporous silica templates have found to be useful in CNT synthesis: Transition metals can be deposited at the pores well-graphitized CNTs can grow from the resulting metal nanoparticles.

Results and Discussion

In this study the catalysts produced were employed in CNT production with the chemical vapor deposition process using acetylene as the hydrocarbon source at different temperatures. Among the many synthetic routes for CNTs, the CVD route is mainly favored because of its scalability and potentially low cost. The catalyst system was placed in to the quartz tube reactor. The system was kept between 500°C and 700°C for 30 min under Ar flow for the stabilization of the catalysts, and then hydrogen gas was passed through the tubular reactor in order to reduce the catalyst into the metallic form. To find the optimum experimental conditions for each catalyst system the parameters (temperature, flow-rate, experiment time) were optimized. After the catalyst system prepared for the production of carbonaceous material, high purity acetylene was started to flow for the formation of the carbon nanostructures. The difference in the morphology, and the structural properties of the CNTs obtained by this method were characterized using SEM, XRD, ^{13}C-NMR and TEM.

Conclusions

In summary, we showed that metal incorporated MCM-41 type catalytic materials were successfully synthesized in 30 minutes by using microwave radiation. The MCM-41 structure was not distorted and the BET surface area values over 1500 m^2/g were obtained having Metal/Si mole ratios over 0.04 while lower values were obtained by impregnation technique. CVD was performed on the MCM-41 based catalysts and carbon nanofiber (CNF) and carbon nanotube formation was detected on Cu and Ni containing species. The concentration seems effective on the success of the CNT/CNF production. Higher Metal:Si ratio catalysts were found to produce more CNT/CNF product.

References

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