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Structural Analysis of Multi-Walled Carbon Nanocoils Synthesized with Fe–Sn Catalyst Supported on Zeolite

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Thin carbon nanocoil (CNC) with a fiber diameter of less than 50 nm was synthesized by catalytic chemical vapor deposition using Fe–Sn catalyst supported on zeolite. The chemical vapor deposition parameters of reaction temperature, gas flow rate of N_2 as dilute gas and C_2H_2 as source gas were 650–750 °C, 1000 ml/min and 50–300 ml/min respectively. Transmission electron microscopic observation revealed that thin CNCs had a hollow and multi-walled structure with cylindrical graphitic layers. More than 90% of the CNCs obtained were multi-walled CNCs (MWCNCs), and the remainder was columnar CNCs without a hollow structure. Three-dimensional images of an MWCNC with Au nanoparticles on its surface were reconstructed by electron tomography and confirmed that the MWCNC had a three-dimensional helical shape.

Keywords: Carbon Nanocoil, Zeolite, Transmission Electron Microscopy, Electron Tomography.

RESEARCH ARTICLE

1. INTRODUCTION

A carbon nanocoil (CNC) is a carbon nanofiber (CNF) with helical shape. CNCs are considered to be potential materials for electron field emitters,[1,2] tactile sensors,[3] nano-springs,[4] electromagnetic wave absorbers,[5] composite sheets[6] and catalyst supports of fuel cells.[7] CNCs are synthesized mainly by catalytic chemical vapor deposition (CVD). Some composite metal catalysts, Fe–Sn,[8–10] Fe-ITO,[1,11] Ni–Sn,[2,12] Ni–Cu,[13] are known to grow CNCs. Helical carbon nanofibers are considered to form owing to the differences in the cracking of hydrocarbon feed stock and segregation rates of carbons between these composite metals. CNCs are also synthesized by adding sulfur to source gas,[14] indicating that the enhancement of carbon segregation is effective.

In carbon nanotube (CNT) synthesis, diameter-controlled single-walled CNTs have been obtained using zeolite as a catalyst supporting material.[15,16] Similarly, the diameter of a CNC has been shown to be proportional to the catalyst particle size.[10] The smaller the catalyst particle size, the thinner the CNC fiber diameter. It has been reported that the coil diameter of a CNC can be as small as 50 nm, in which case the CNC was grown from ≈50 nm diameter catalyst particles.[9] Thin CNCs were also synthesized with Ni–Fe[17] or Co[18] catalyst supported on mesoporous silica and Fe–Co catalyst supported on MCM-41-type mesoporous silicates.[19] These reports tell us that a catalyst particle with nanometer scale can synthesize thin CNCs with a fiber diameter of less than 50 nm. To understand the growth mechanism of thin CNCs, analysis of their three-dimensional (3D) structure is necessary.

In this study, Fe–Sn catalyst particles supported on zeolite were used to synthesize thin CNCs, where the catalyst particle size was controlled using a simple chemical process. The structures of thin CNCs were analyzed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). A 3D tomography system equipped with a transmission electron microscope can reconstruct 3D images of thin CNCs. The structure of thin CNCs including their helical direction was clarified from the 3D images.

2. EXPERIMENTAL DETAILS

Thin CNCs were synthesized using a catalytic CVD method and Fe–Sn catalyst supported on Y-type zeolite

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