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Revisited Roles of Bimetallic Catalysts for Controlled CVD Growth of Single-Walled Carbon Nanotubes

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Abstract Text:

Bimetallic catalysts such as Co-Mo have been used for efficient growth of vertically aligned single-walled carbon nanotube (SWNTs) for a decade [1]. Recently, different kinds of bimetallic catalyst such as Co-W [2] or Co-Cu [3] are employed for structure controlled growth of SWNTs. Different roles of bimetallic catalysts are revisited by newly proposed in-plane transmission electron microscopy (TEM) technique, which enables a direct TEM characterization of catalysts and CVD grown nanotubes on SiO_2 TEM grid. Sputtered and dip-coated cobalt-based catalysts, i.e., Co, Co-Mo, Co-Cu, Co-W are used in alcohol catalytic CVD (ACCVD).

We found the Co oxide catalysts can efficiently grow narrower diameter and smaller average diameter SWNTs compared with pre-reduced Co catalysts. The in-plane TEM and X-ray photoelectron spectroscopy (XPS) reveal that Co catalysts are transformed to Co3O4 after reduction-calcination process and then decompose to CoO before growth at a typical growth temperature (800 ºC) in Ar atmosphere. We conclude that an in-situ reduction process occurred on low-mobility CoO after the introduction of ethanol is essential to activate small metallic Co catalysts. The in-plane TEM studies confirmed that Co-Mo bimetallic catalysts have the same mechanism as oxidized Co system, i.e. low mobility oxides and reduced metal, consistent with our previous report [1].

By using Co-Cu catalysts, we can synthesize vertically aligned SWNTs with subnanometer diameters on quartz (and SiO2/Si) substrates [3]. Scanning transmission electron microscopic energy-dispersive X-ray spectroscopy (EDS-STEM) and high angle annular dark field (HAADF-STEM) imaging of the Co/Cu bimetallic catalyst system showed that Co catalysts were captured and anchored by adjacent Cu nanoparticles, and thus were prevented from coalescing into larger size, which contributed to the small diameter of SWNTs.

High-melting point W6Co7 alloy is reported to grow a single chirality, (12,6) with over 90 % abundance through high-temperature (1030 ºC) reduction and growth [2]. Here, we show that a sputtered Co-W catalyst can selectively grow (12,6) SWNTs by CVD at lower temperatures. Statistical Raman mapping analysis and optical absorption spectrum of the as-grown SWNTs reveal that the abundance of (12,6) is over 50%. The morphology and structure of catalyst is investigated by the in-plane TEM, which discloses the complicated structure changes before and after growth.

References:

[1] M. Hu, Y. Murakami, M. Ogura, S. Maruyama, T. Okubo, J. Catalysis, 225 (2004) 230.

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[3] K. Cui, A. Kumamoto, R. Xiang, H. An, B. Wang, T. Inoue, S. Chiashi, Y. Ikuhara, S. Maruyama, Nanoscale, (2015) [DOI: 10.1039/C5NR06007A].

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