Purification and Characterization of Single-walled Carbon Nanotubes Synthesized by Catalytic CVD on Zeolite from ethanol

Hideyuki Igarashi,^a Hiroto Murakami,^a Yoichi Murakami,^b Shigeo Maruyama,^b and <u>Naotoshi</u> <u>Nakashima</u>^c*

^a Department of Materials Science, Graduate School of Science and Technology, Nagasaki University, Bunkyo, Nagasaki 852-8521, Japan

^o Department of Mechanical Engineering, The University of Tokyo, Bunkyo-ku,

Tokyo 113-8656, Japan

^c Department of Chemistry and Biochemistry, Graduate School of Engineering, Kyushu University, Hakozaki 6-10-1, Fukuoka 812-8581, Japan

Phone/FAX: (+81)-92-642-3602

e-mail:nakashima-tcm@mbox.nc.kyushu-u.ac.jp

Carbon nanotubes have been synthesized by laser ablation, arc-discharge and chemical vapor deposition (CVD). Catalytic CVD (CCVD) has been developed in recent several years for large-scale production and controlled synthesis of single-walled carbon nanotubes (SWNTs) [1,2]. Shinohara et al. reported large-scale production of quasi-aligned multi-walled carbon nanotube bundles on zeolite by CCVD, in which acetylene was used as the carbon source to produce nanotubes by catalytic decomposition [3]. High-quality SWNTs were produced by Maruyama et al. on zeolite from alcohol by CCVD, where oxygen radical generated from alcohol was suggested to contribute production of SWNTs by attacking carbon impurities such as amorphous carbon [4]. CCVD synthesis and purification of SWNTs have been reported on aerosol-supported also catalyst[5], or from CO disproportionation over Co-Mo catalyst using silica support [6,7].

We describe a method for purification of zeolite-supported single-walled carbon nanotubes (SWNTs) catalytically synthesized by chemical vapor deposition using ethanol as the carbon source. The following procedures were found to be effective for nearly complete removal of zeolite particles and Fe/Co catalysts from raw SWNTs: i) heating of raw material at 240 °C for 18 h or 340 °C for 1.5 h, ii) treatment with 1 % aqueous solution of hydrofluoric acid for at least 30 min, and iii) drying at 350 °C for 1h. The yields of SWNTs were $\approx 95\%$ and the purities of SWNTs thus obtained were more than 95 %, as characterized by thermo-gravimetric (TG) analysis. The raw and purified SWNTs were also characterized by EDX spectroscopy and transmission electron microscopy (TEM).

References

- M.J. Bronikowski, P.A. Willis, D.T. Colbert, K.A. Smith, R.E. Smalley, *J. Vac. Sci. Technol.* A 19, 1800(2001).
- [2] Y. Zhang, A. Chang, J. Cao, Q. Wang, W. Kim, Y. Li, N. Morris, E. Yenilmez, J. Kong, H. Dai, *Appl. Phys. Lett.* **79**, 3155(2001).
- [3] K. Mukhopadhyay, A. Kosho, T. Sugai, N. Tanaka, H. Shinohara, Z. Konya, J.B. Nagy, *Chem. Phys. Lett.* 303,117 (1999).
- [4] Y. Murakami, Y. Miyauchi, S. Chiashi, and S. Maruyama, *Chem. Phys. Lett.* **374**, 53(2003).
- [5] B. Zeng, Y. Li, J. Liu, Appl. Phys. A ,74,345 (2002).
- [6] W.E. Alvarez, F. Pompeo, J.E. Herrera, L. Balzano, D.E. Resasco, *Chem. Mater.* 14, 1854 (2002).
- S.M. Bachilo, L. Balzano, J.E. Herrera, F. Pompeo, D.E. Resasco, R.B. Weisman, J. Am.
- *Chem. Soc.* **125**,11186 (2003).