

Monday Morning, November 4, 2002

Nanotubes: Science and Applications Topical Conference Room: C-209 - Session NT-MoM

Nanotubes: Growth and Characterization

Moderator: S.B. Sinnott, University of Florida

8:20am **NT-MoM1 Synthesis and Applications of Vertically Aligned Carbon Nanofibers**, *V.I. Merkulov, A.V. Melechko, M.A. Guillorn, D.K. Hensley, D.H. Lowndes, M.L. Simpson*, Oak Ridge National Laboratory
INVITED

Vertically aligned carbon nanofibers (VACNFs) prepared by direct-current (dc) plasma enhanced chemical vapor deposition (PECVD) are important for various applications including electron field emitters, tips for scanning microscopy, and biological probes, among others. To date, the crucial advantage of using VACNFs is the ability to grow them deterministically, i.e. their location, height, tip and base diameters, and, to some extent, shape, orientation, and chemical composition can all be controlled, and mechanically and electrically reliable contact to the substrate can be established. We will discuss various aspects of the VACNF growth by dc PECVD including the effects of the growth parameters on the properties of resultant VACNFs, the VACNF alignment, etc. We will demonstrate significant progress toward the ability to deterministically synthesize VACNF-based carbon nanostructures in a large-scale synthesis process. In addition, phenomenological models that explain important aspects of VACNF growth will be presented. Finally, various potential applications of VACNFs will be discussed, and the possibility of large-scale fabrication of several types of devices based on individual VACNFs will be demonstrated.

9:00am **NT-MoM3 Facilitating Single-wall Nanotube Formation by Plasma Excitation**, *B. Simard, C.T. Kingston, S. Denommee, D. Ruth*, National Research Council Canada

We have investigated the effects of laser excitation of the ablation plasma on single-wall nanotube (SWNT) formation using the laser-oven method. In the first study a high-power kilohertz YAG laser was used to thermally excite the target and ablation plasma. A sustainable production rate of 125 mg/hr of very pure SWNT material has been achieved. A second study involved the use of an argon-ion laser to electronically excite the G_0 molecule via the swan-band transition. The results with respect to nanotube yield, purity and characteristics for both studies will be presented.

9:20am **NT-MoM4 Directed Growth of Carbon Nanotubes on Anodized Aluminum Surfaces**, *J.D. Whittaker, M. Housley, W. Wilson, R.C. Davis*, Brigham Young University

Aluminum oxide surfaces were prepared from 20 nm aluminum films by anodic oxidation. The anodization was performed in a solution of tartaric acid and propylene glycol yielding smooth alumina surfaces. Iron was then deposited by thermal evaporation, in varying thicknesses, to act as a catalyst. Carbon nanotubes were grown on the surface by chemical vapor deposition, and characterized using scanning electron microscopy and atomic force microscopy. We will also discuss NT growth from ordered porous alumina films. These growth methods are being developed to control the location and orientation of tubes grown on solid surfaces.

9:40am **NT-MoM5 A Novel CVD Method for Large-scale Synthesis of Carbon Nanotubes**, *B. Simard, S. Denommee, Z. Yang, D. Ruth, C.T. Kingston*, National Research Council of Canada, *N. Braidy, G. Botton*, McMaster University, Canada

A novel CVD method for large scale synthesis of carbon nanotubes is demonstrated. It relies on the "in-situ" formation of metal nanoparticles into the feedstock solution, which is then vaporized from an aerosol nozzle into a furnace maintained between 800-1000 C. The nanoparticles are formed from laser vaporization of the bulk materials, either Ni/Co 50/50 or Co/Mo 50/50 directly in the feedstock. The nanoparticles are produced with a very narrow distribution of less than 1 nanometer with average size smaller than 3 nm. The technique is fully scalable. TEM of the synthesized nanotubes and the nanoparticles has been performed and the results will be shown at the meeting as well as more detailed description of the apparatus.

10:00am **NT-MoM6 Preparation, Chemical and Physical Properties of Large Area Free-Standing Carbon Nanofilms**, *M.M. Kappes*, Universität Karlsruhe, Germany

Thin free standing films of single-walled carbon nanotubes (SWNT) were prepared by a novel ultrafiltration process. 1 cm x 1 cm films of less than 200 nm thickness can be routinely prepared for a variety of SWNT

materials ranging from as-prepared to chemical derivatized (via amide coupling). Such films are particularly useful for transmission spectroscopic probes. We present results on gas-adsorption leading to changes in electronic structure. Furthermore we have investigated changes to optical properties associated with thermally induced deintercalation of SWNT bundles. Such processes may also be induced via visible laser irradiation thus allowing for spatial resolution.

10:20am **NT-MoM7 Growth of Aligned Arrays of Carbon Nanotubes from Patterned Catalyst Particles**, *M.J. Bronikowski, D.S. Choi, M.E. Hoenk, R.S. Kowalczyk, F. Noca, M.E. Taylor, R.M. Williams, E.W. Wong, B.D. Hunt*, Jet Propulsion Laboratory

Carbon nanotubes (CNT) are expected to have a wide variety of uses due to their many exceptional properties. In particular, regular arrays of CNT are expected to have applications ranging from biomolecular filters to nano-scale electronics, oscillators, and signal processors. Of central importance in such applications is the ability to grow arrays of CNT with identical dimensions and tube-tube spacings. This talk will discuss our recent efforts in JPL's Microdevices Laboratory toward this goal. CNT are grown from gaseous carbon-containing precursors (hydrocarbons, CO) on substrate surfaces such as Si or SiO₂ by chemical vapor deposition (CVD): CNT nucleate and grow from nanometer-size particles of catalytic metals (Fe, Ni, Mo) that have been pre-deposited on the substrate. CNT dimensions and arrangement are determined by the size and arrangement of the catalyst dots, and by the CVD growth parameters. Various methods for controllably placing catalyst particles on surfaces will be demonstrated and discussed. In particular, methods for patterning catalytic metals with nanometer precision using self-assembling thin films of block copolymers will be presented.

10:40am **NT-MoM8 Carbon Nanotube Growth for Nanomechanical Devices**, *M.E. Hoenk, R.S. Kowalczyk, M.J. Bronikowski, E.W. Wong, D.S. Choi, F. Noca, R.M. Williams, M.E. Taylor, B.D. Hunt*, Jet Propulsion Laboratory, California Institute of Technology

Mechanical resonators with nanometer dimensions offer the capability for sensors and actuators to interact with materials at the molecular scale. We are developing device structures based on carbon nanotube mechanical resonators. While the unique mechanical and electronic properties of carbon nanotubes are promising for this application, the relationship between growth conditions and nanotube properties plays a major role in nanotube device development. In this paper, we will present our most recent results on the growth, processing, and characterization of carbon nanotube arrays. We have grown aligned carbon nanotube arrays over a wide range of pressures and temperatures using thermal and plasma-enhanced chemical vapor deposition. We have experimented with a variety of catalyst materials, growth conditions, and patterning techniques, and we have characterized the catalysts and nanotubes using scanning and transmission electron microscopy, Raman spectroscopy, and atomic force microscopy. We have demonstrated growth of aligned arrays of carbon nanotubes at temperatures as low as 411 C. The research described in this paper was performed at the Jet Propulsion Laboratory, California Institute of Technology, and was jointly sponsored by the National Aeronautics and Space Administration, Office of Aerospace Technology, and the Defense Advanced Research Projects Agency, Microsystems Technology Office.

11:00am **NT-MoM9 High-resolution Transmission Electron Microscopy Study of Catalyst Metal Particle at the Tip of Carbon Nanotube**, *T. Ikuno, S. Takahashi, K. Kamada, S. Ohkura, M. Katayama, T. Hirao, K. Oura*, Osaka University, Japan

Carbon nanotubes (CNTs) have been attracted much attention due to their fundamental research interest and potential applications. Control of alignment of CNT is essential for applications such as field emission display (FED), quantum wire, and field effect transistor. For FED application, to concentrate electric field at the CNT tip, vertically aligned CNT is desirable. On the other hand, lateral alignment is necessary for nanodevice application. In this study, we have performed high-resolution transmission electron microscopy (TEM) analysis focused on the metal particle at the tip of CNT to clarify correlation between growth mechanism of CNT and crystallography of metal catalyst. Specimens are randomly and vertically aligned CNTs which are synthesized on Ni (50 Å)/ Si (100) substrates by thermal chemical vapor deposition and RF-plasma CVD, respectively. From TEM observation of Ni particle at the tip, it was found that randomly CNT (bamboo-like CNT) has spherical Ni particle at the tip, and vertically CNT (multi-wall CNT) has a wedge shape with flat facets. Both particles were found to be monocrystalline. The crystalline orientations of the particles were also investigated. The axial directions of the vertically CNT are mainly parallel to the <111> and <311> direction of

Ni. From high-resolution TEM image of Ni / CNT interface, graphite layers were formed along the ridge line of Ni particle for randomly CNTs, whereas they were formed along specific planes parallel to the growth direction for vertically CNTs. On the basis of these results, we will discuss the mechanism of alignment process associated with growth front of graphite layers precipitated from the Ni particle. This work was performed with Japan Fine Ceramic Center under the Frontier Carbon Technology Project of the New Energy and Industrial Technology Development Organization.

11:20am **NT-MoM10 Nitrogenated Carbon Nanostructures Grown by Microwave Induced Hot-filament CVD Techniques**, *D. Sarangi, A. Karimi*, FSB-IPMC, EPFL, Switzerland

Nitrogenated carbon nanotubes and nanostructures were grown by microwave induced hot-filament chemical vapor deposition (CVD) technique. The mixture of methane or acetylene gas with ammonia or nitrogen gas was used as feed gas. The nanostructures were grown on silicon substrates using iron (Fe) and nickel (Ni) as catalysts. The morphological properties of the nanotubes and nanostructures were studied using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The effect of microwave power and the influences of precursor gases on the structures of nanotubes were discussed. About 5 atomic percentage of nitrogen was observed as measured by electron energy loss spectroscopy (EELS). High-resolution TEM observation revealed that, with the increase of nitrogen concentration the outer graphane shell of the nanotube becomes more rough, retaining the crystallinity. In general, this paper will discuss the morphological properties of the carbon nanostructures with respect to the mechanical properties.

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