

Wednesday Morning Poster Sessions, November 5, 2003

Nanotubes

Room Hall A-C - Session NT-WeP

Poster Session

NT-WeP1 Reactor Design Considerations in the Hot Filament/DC Plasma Synthesis of Carbon Nanotubes/Nanofibers, B.A. Cruden, A.M. Cassell, Q. Ye, Eloret Corporation/NASA Ames Research Center; M. Meyyappan, NASA Ames Research Center

A combined hot filament/DC plasma approach to chemical vapor deposition (CVD) of carbon nanofibers (CNFs) has been explored. As a part of the study, the impact of filament usage and substrate holder design has been examined by SEM imaging of deposition products and monitoring of downstream products by residual gas analysis (RGA). It is demonstrated that the filament wire is important only in the pre-treatment of the substrate, where ammonia is dissociated and reacts with the substrate surface, improving CNF growth quality. However, the filament has no apparent impact when combined with the DC plasma, as demonstrated by RGA analysis of the plasma chemistry and the resultant films. The substrate holder is modified by introducing a graphite spacer into the electrode. By varying the size of the spacer, the effective surface.

NT-WeP2 Large-Area Growth of Aligned Carbon Nanotubes by Hot-Filament Assisted DC Plasma CVD, K. Ueda, T. Negishi, Y. Hayashi, S. Nishino, Kyoto Institute of Technology, Japan

Hot-filament assisted dc plasma chemical vapor deposition (HF/DC-PCVD),@footnote 1@ in which a plasma is generated between the anode of hot-filaments and the cathode of a substrate, was developed to grow carbon nanotubes in a large-scale. Carbon nanotubes are expected to be used for the field emitters of a field emission display (FED) because their high aspect ratio and small radius of curvature lead to large electric-field enhancement at their tips resulting in low operating voltage for electron emission. However the method of large area growth of vertically aligned carbon nanotubes should be developed to be applied to field emitters of FED. We have succeeded to grow well-aligned carbon nanotubes on an iron substrate of 5 X 5 cm@super 2@ in a CH@sub 4@/H@sub 2@ DC plasma at 2.7kPa (20Torr) by HF/DC-PCVD. In this process, the plasma sheath plays an important role for the growth of vertically aligned carbon nanotubes. The sheath was formed all over the substrate with uniform thickness, i.e., uniform electric field strength, when three hot-filaments spaced 5mm were stretched parallel to the substrate. In order to grow well-aligned carbon nanotubes on a larger substrate, we generated plasma above a substrate of 10 X 10 cm@super 2@ at 1.35kPa (10Torr) using three hot-filaments spaced 15mm. Therefore the large-area growth of well aligned carbon nanotubes on a 10 X 10 cm@super 2@ substrate is expected. @FootnoteText@ @footnote 1@Y.Hayashi, T.Negishi, and S.Nishino, J. Vac. Sci. Technol. A 19(2001) 1796.

NT-WeP3 Carbon Nitride Nanostructures Prepared by Surface Treatments, J.H. Hong, S.H. Kim, S. Lee, Y.B. Hahn, Chonbuk National University, Korea

Nano-dots and nano-stripes of carbon nitride (CNX) were fabricated by surface treatment. Amorphous CNX thin films grown on Si (100) wafer by plasma enhanced chemical vapor deposition at room temperature were first treated by H₂ plasma and then annealed at 200-300@degree@. The effects of surface treatments on chemical and structural properties of the films were investigated by Auger electron spectroscopy (AES), Fourier transform infrared spectra (FT-IR). Well-defined nano-dots and nano-stripes were formed depending on temperature and treatment time. This was attributed to recombination of adatoms and rearrangement of film structure caused by surface energy change between the film and the substrate. The AES showed that the composition of the film is 90 at.% C and 9 at.% N. The FT-IR spectra showed the presence of C-N peak at 1260 cm⁻¹ and C=N peak at 1640-1670 cm⁻¹, respectively.

NT-WeP5 Preparation of Self-Assembled Carbon Nano-Ropes and Carbon Nanotubes Using Microwave Plasma-Enhanced Chemical Vapor Deposition@footnote 1@, M. Taniguchi, M. Hiramatsu, Y. Ando, Meijo University, Japan; M. Hori, Nagoya University, Japan

Carbon nano-structures such as carbon nanotubes, nanofibers and nanocrystalline diamond films are of tremendous interest from both a fundamental and an applied prospective. From the point of view of their wide applications, it is desirable to control properties such as the size, shape, and growth direction of surface structures during the growth.

Carbon nano-structures with different structure and morphology can now be fabricated using several techniques. Among various techniques used for the growth of carbon nano-structures, plasma-enhanced chemical vapor deposition (PCVD) has gained considerable importance for the industrial application due to its feasibility and potentiality for large-area production with reasonable growth rates. In this work, aligned carbon nano-structures were grown using a conventional microwave plasma-enhanced chemical vapor deposition (MWPCVD) with a 1.5-kW microwave generator. A mixture of acetylene and hydrogen was used as a carbon source gas. The applied microwave power and the pressure during the growth were 400-1200 W and 25-80 Torr, respectively. The growth experiments were carried out for 1-15 min at substrate temperatures ranging from 500 to 800 °C. The morphologies of the grown carbon nano-structures depended strongly on the growth temperature. Vertically aligned, self-assembled, stranded carbon nano-fibers (carbon nano-ropes) were grown at relatively low temperature of about 500 °C. On the other hand, carbon nanotubes were grown on the catalyzed Si substrate at the temperature of about 800 °C. The field emission characteristics for the aligned carbon nano-structures were investigated. @FootnoteText@ @footnote 1@This work was supported by 21st century COE program, Nano Factory.

NT-WeP6 Synthesis of Multi-walled Carbon Nanotubes by CVD using Methane and Acetylene, S.A. Moshkalyov, UNICAMP, Brazil; C. Reyes-Betanzo, INAOE, Mexico; A.C.S. Ramos, J.L. Gonçalves, J.W. Swart, UNICAMP, Brazil

For the CNTs growth, a number of methods was developed, including different versions of chemical vapor deposition (CVD). CVD methods have certain advantages over other ones as they provide a way for controlled, directional growth of both single-walled and multi-walled CNTs. Here, results of CNTs synthesis using two different catalytic CVD techniques (plasma-enhanced low-pressure CVD and atmospheric-pressure thermal CVD) are presented. Thin Ni films (1-40 nm thick) were used as a catalyst material. For the film deposition, electron-beam thermal deposition was employed. Then, the films were thermally treated (~700 C) in a nitrogen atmosphere to provide formation of separate catalyst nanoparticles. As substrates, Si wafers were used previously covered by thin (50 nm) oxide films. CNTs synthesis was realized in two different reactors. The first one uses a low-pressure microwave plasma source, with low-pressure (~1Torr) nitrogen-acetylene gas mixtures. Samples are heated up to 700 C by a halogen lamp heater. In the second reactor, the flowing gas mixture and samples are heated in a resistive heating furnace to temperatures up to 900 C. Atmospheric pressure methane-hydrogen based mixtures were used in this case. After synthesis, the samples were examined using high-resolution scanning electron microscopes. The first results obtained here have shown fast CNT growth in both reactors. The process appears to depend critically on the catalyst thickness. For thin catalyst films, it was possible to grow long (randomly oriented), and small diameter (smaller than 10 nm) CNTs. In most cases, small catalyst particles were detected at the tip of the tubes. This suggests that the tip growth mechanism is responsible for the CNTs synthesis under the present conditions. The future work will focus on comparison of the two techniques and optimization of the processes, in particular, in order to achieve directional CNTs growth.

NT-WeP7 Growth of Cone-shaped Carbon Nanotubes by Arc Discharge in Anode, S.-C. Kung, Industrial Technology Research Institute,Taiwan; C.-M. Hsu, Industrial Technology Research Institute,Taiwan, Taiwan; B.-J. Li, H.-J. Lai, Industrial Technology Research Institute,Taiwan

Various synthetic methods are developed and the main method for the production of high quality CNTs (carbon nanotubes) is the DC (direct current) arc discharge between two graphite electrodes in a buffer atmosphere which is usually is helium, leading to the formation of nanotube-containing deposit on the end of the cathode. Many factors such as the velocity distribution of carbon ions, the density of carbon vapor, and the growth temperature have been suggested to affect the growth of CNTs. The pressure of carrier gas in the evaporation chamber is generally considered as the most important factor. Here, we report the production of CNTs without catalytic metals by DC arc discharge between two graphitic rods of the same diameter at the low pressure ambience (ca. 10@super - 2@ torr) and higher pressure helium atmosphere (ca. 900 torr). After the arcing was carried out, CNTs were discovered in the deposit on the anode instead of the cathode in low pressure working ambience. The morphologies of CNTs on the anode deposit are specifically cone-shaped, 15 nm in diameter. The difference of nanometric graphitic structures synthesized under the noble and higher vacuum atmosphere were discussed and characterized by FESEM, HRTEM, RAMAN spectrometer, TGA, and field emission properties measurement.

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NT-WeP8 Growth of Carbon with Vertically Aligned Nano-scale Flake Structure by rf Sputtering, E. Kusano, H. Zhang, Kanazawa Institute of Technology, Japan; T. Kogure, University of Tokyo, Japan; I. Yoshimura, K. Yamamoto, A. Kinbara, Kanazawa Institute of Technology, Japan

Carbon nanoflake has been deposited by rf sputtering using the mixture of Ar and CH₄ with a total pressure of 14.5Pa as a discharge gas and a graphite disk as a target. A Si (110) wafer was used as a substrate on which carbon nanoflakes was grown at 670°C. The rf (13.56MHz) discharge power was kept 100W for all deposition. Deposition time was changed from 15 min to 3 hours. Microstructure of deposited carbon was investigated by Field Emission Scanning Electron Microscope (FESEM) and High Resolution Transmission Electron Microscope (HRTEM). Under the present conditions, high-density vertically aligned carbon nanoflakes with a thickness of about 30 nm were obtained. High intensity and symmetry of electron diffraction pattern indicated that carbon nanoflakes deposited by rf sputtering had the three dimensionally perfect crystallinity with an interlayer spacing of 335pm. In particular, there was no disorder in stacking of layer structure. It was further found that the thickness of the flakes was independent of deposition time while the length of the flakes increased to about 600-800 nm with increasing deposition time to 3 hours. Width of the carbon nanoflake was about 300 nm after 3-hour deposition. The results suggest that a critical thickness of carbon nanoflake exists, possibly depending on deposition conditions such as substrate temperature and discharge pressure. This critical thickness was almost equal to the layer number of about 90. Some potential applications of the carbon nanoflake film are being considered in vacuum electronic devices, chemical catalyst, frictional abrasion resistance, etc.

NT-WeP9 Synthesis of Aluminosilicate Nanotube "Imogolite" in the Presence of Polymer Solution, K. Yamamoto, H. Otsuka, S.-I. Wada, A. Takahara, Kyushu University, Japan

Inorganic nanotube "Imogolite" is a hydrous aluminosilicate polymer with an external diameter of ca. 2.5 nm and lengths from several hundreds nanometer to a micrometer. The authors introduce a novel method for the preparation of polymer nanohybrid through in situ synthesis of imogolite in the aqueous solution of water-soluble polymer. A polymer nanohybrid prepared by this method can be expected to improve the dispersion of imogolite in the polymer matrix compared with that prepared by conventional blending. The aqueous dilute solution of aluminum chloride and tetraethoxysilane was stirred and refluxed at 369 K for several days and freeze-drying of the solution gave white powder of synthetic imogolite. The polymer nanohybrid was also prepared through in situ synthesis of imogolite in the presence of poly(vinylalcohol)(PVA) in solution and this reaction was carried out at various concentration of PVA. The formation of synthetic imogolite was confirmed by IR, WAXD measurement, and AFM observation. The content of imogolite in the each polymer hybrid sample was evaluated by TGA measurement. WAXD profiles of PVA-imogolite nanohybrid showed the sharp diffraction peak of PVA and the diffractions corresponding to the parallel orientation of the bundle of imogolite nanotubes. It was confirmed that synthetic imogolite formed nanoscale fibrous network structure in PVA matrix by the above mentioned procedure. Furthermore, high transparency of synthetic imogolite/PVA hybrid film prepared by this method was attained because of the high dispersibility of synthetic imogolite in PVA solution. @FootnoteText@ @footnote 1@K. Yamamoto, H. Otsuka, S. -I. Wada, A. Takahara, J. Adhesion, 78, 591 (2002).

NT-WeP11 A Novel Field Emission Triode Configuration Based on a Cylinder/Plane Geometry and Carbon Nanotubes, V.P. Mammanna, O. Shenderova, G.E. McGuire, International Technology Center

Field emission devices are an excellent alternative for a myriad of applications requiring highly efficient and compact electron sources, such as flat panel displays, microwave tubes and plasma thrusters. Although field emission devices with several different triode configurations have been developed, there is still a need for devices with increased robustness, lower gate voltage and lower gate parasitic current that are easy to manufacture. In this work we propose a new configuration based on a cylinder/plane geometry, in which the cylinder plays the role of cathode and the plane performs as the gate. In the proposed configuration, an insulating dielectric is used between the cathode and the gate, in a way that the gate current is significantly reduced while the overall robustness is increased. Manufacturing this structure is straightforward, since it does not require patterning of the dielectric. The complexity is further reduced if no pixel addressing is needed (for non-display applications), since the dielectric may be blanket deposited or grown. We present electrostatic field calculations made in order to optimize the geometrical parameters of

the device, and these calculations demonstrate that emission takes place at gate potentials of a few tens of volts, if carbon nanotubes are included into the structure in order to enhance the local electrostatic field. In this paper we address the dependence of the electrostatic field on the gate voltage, type of dielectric, cylinder/plane distance and cylinder cross-section. Some focusing issues are preliminarily addressed too. A process flow for the manufacture of the device is also proposed.

NT-WeP12 Field Emission Properties of Carbon Nanotubes Relative to Buffer Layer, C.-M. Hsu, S.-C. Kung, B.-J. Li, H.-J. Lai, Industrial Technology Research Institute, Taiwan

Effect of field emission and growth of carbon nanotubes use various buffer layers Carbon nanotubes (CNTs) were synthesized with various buffer layers by electron cyclotron resonance chemical vapor deposition (ECR-CVD) and microwave plasma chemical vapor deposition (MPCVD) system. Growth was performed in a flowing mixture of H₂, CH₄ and N₂ as precursors at temperature 600°C and -200 V substrate bias, sputter Co as catalysts material. The preliminary results indicate that well-aligned carbon nanotubes show significant emission current. Field emission property of carbon nanotubes and buffer layers relationship will be compared. Field emission scanning electron microscopy (FESEM) shows that the CNTs are well aligned with high aspect ratio and growth direction vertical to the substrate. The field emission properties of CNTs and buffer layers relationship will be characterized by I-V measurement, XRD, TEM, and RAMAN.

NT-WeP14 Modification of Contact Resistance by Nano-bonding and Thermal Treatment, S. Lee, H.J. Chung, CSNS and Seoul National University, Korea; J.-Y. Park, Cornell University; Y.S. Cho, Y.D. Park, J.H. Choi, Y. Kuk, CSNS and Seoul National University, Korea

When we connect a nano-structure with conducting wires, we face difficulty fabricating them around ultra-fine and dense nanostructures. In order to use nanotubes or nanowires as interconnection materials, the contact resistance should be controllable because it may become main noise and dissipation sources with this low electrical current signal. In this study, the variation of the contact resistance in the carbon nanotubes (2-8 nm) on Au electrodes was investigated. The contact resistance between a nanotube and an electrode was changed the nanometer sized dots of Au and Ni formed by the field evaporation of the metals coated cantilever using atomic force microscopy (AFM) combined with a thermal treatment of 400-500 °C by a rapid thermal annealing (RTA). By using this method, the contact resistance of ~100 kΩ is routinely produced despite of the geometry of nanotubes. This method can be used to other transport experiments to be required to modify the contact resistance.

NT-WeP15 Fabrication and Characterization of Active Nanoscale Electronic Devices Based on Vertically Aligned Carbon Nanofibers (VACNF), M.L. Simpson, Oak Ridge National Laboratory; X. Yang, University of Tennessee; M.A. Guillorn, Oak Ridge National Laboratory; D.W. Austin, University of Tennessee; V.I. Merkulov, A.V. Melechko, D.H. Lowndes, Oak Ridge National Laboratory

Arrays of deterministically grown VACNFs were synthesized in a high-density Plasma-Enhanced Chemical Vapor Deposition (PECVD) process that provides a high degree of control of the growth conditions and, consequently, the resultant electronic properties. After inspection of fiber morphology and composition using scanning electron microscopy (SEM) and energy dispersive x-ray (EDX) analysis, a layer of SiO₂ was deposited conformally onto the fibers by a silane-based RF PECVD process. Following planarization of the substrates by chemical mechanical polishing, the tips of the fibers were uncovered by reactive ion etching of the SiO₂ layer. Electrodes were patterned on the substrate surface to make contact to individual fiber tips within the array. Current versus voltage (I-V) curves indicate the presence of a metal-semiconductor (Schottky) junction and display a rectifying behavior. I-V measurements made with respect to temperature reveal an average barrier height of 300 mV. Additional experiments indicate that the junction occurs at the nanofiber/silicon interface. An empirical model of the material system will be presented along with an explanation of the experimentally observed charge transport behavior.

NT-WeP16 Fabrication of Top gated Single-Walled Carbon Nanotube Field Effect Transistor Utilizing Scanning Probe Lithography, S.H. Choi, J. Heo, SungKyunKwan University, Korea; W.B. Choi, Samsung Advanced Institute of Technology, Korea; I.S. Chung, SungKyunKwan University, Korea

We attempted to fabricate top gated single wall carbon nanotube field effect transistor (SWNT FET) utilizing scanning probe lithography (SPL)

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method. In SPL, electrons induce chemical reactions at the desired position during scanning. Thus, gate electrode as well as source/drain formation would be done using lift-off method without using e-beam lithography. N type or p type FET can be formed according whether the oxygen annealing process is given or not. Thus, we can easily form an inverter based on top gate structure. In this study, SWNTs were dispersed on pre-patterned substrate prior to deposit top gate oxide. Our device shows excellent electric characteristics by thinner gate oxide.

NT-WeP17 Large Paramagnetic Susceptibility and Field Induced Persistent Current for Carbon Nanotube Tori, R. Tamura, Shizuoka University, Japan; *M. Tsukada,* University of Tokyo, Japan

Magnetic susceptibilities have been calculated for a number of Carbon nanotube (CNT) tori with the tight binding model. We found large paramagnetic susceptibility which cannot be expected when the system size is larger than the order of the nanoscale. The CNT torus with six-fold rotational symmetry is composed of six unit cells. The unit cell is an armchair nanotube with two pentagonal defects and two heptagonal defects. Direction of the tube axis is changed by sixty degrees by the defects. When this unit cell is repeated periodically, on the other hand, the helical CNT is formed. The energy levels of the CNT torus can be obtained from the dispersion relation of the helical CNT, $E(k)$, by choosing only the discrete wave number $k = \pi j/3$ with an integer j . The magnetic field penetrating the torus surface change the dispersion relation $E(k)$, while magnetic field through the hole shift the discrete wave number due to the AB effect. When the helical CNT is semi-metallic, it is possible that LUMO band bottom at $k=0$ is lower than the HOMO band over some k region. In that case, electron at the HOMO band makes a transition to the $k=0$ LUMO state so that number of occupied levels with positive k is different from that with negative k . This causes the persistent current and large positive magnetic susceptibility, in spite of large negative magnetic susceptibility of graphite. *Tamura and M. Tsukada, Journal of Physical Society of Japan, Vol. 68, pp.910-922 (1999); K. Akagi, R. Tamura, M. Tsukada, S. Itho, and S. Ihara, Phys. Rev. B, Vol. 53, pp.2114-2120 (1996).*

NT-WeP19 Experimental and Theoretical Studies on the Ozone Reactivity with Carbon Nanotubes, S. Picozzi, L. Lozzi, F. Di Gregorio, S. Santucci, C. Cantalini, INFN and University of L'Aquila, Italy; *C. Baratto, G. Sberveglieri,* INFN and University of Brescia, Italy; *L. Valentini, I. Armentano,* University of Perugia, Italy; *B. Delley,* Paul Scherrer Institut, Switzerland

In a previous paper it has been reported how carbon nanotubes (CNTs) thin films deposited by plasma-enhanced chemical vapor deposition have a strong reactivity with NO₂. In this work we investigate a CNT film as resistive gas sensors for O₃. The sensor composed by the aligned CNT film with a thickness of 200 nm exhibits sensitivity to O₃ gas at concentrations as low as 25 ppb, fast response time with a baseline drift that has been observed if the operating temperature of the sensor is increased over 70°C. Upon the reaction with O₃, the electrical resistance of the CNTs is found to decrease. In order to obtain a theoretical validation of the experimental results, the equilibrium position, charge transfer and density of states are calculated from first principles for the CNT+O₃ system within the density functional theory, using the all-electron DMol3 (density functional theory for molecules and three-dimensional periodic solids) code. Our calculations show that the ozone molecule adsorbs on the tube with a binding energy of the order of 300 meV and gains about 0.1 electrons from the CNT. The calculated density of states shows that O₃ adsorption gives rise to an acceptor peak at the Fermi level, which lies in correspondence to the tube valence band maximum, rendering the CNT+O₃ system metallic. This is consistent with the experimentally observed increase in conductivity. The baseline shift of the sensor experimentally observed for the higher working temperatures may be ascribed to a consumption of the carbon nanotube under the ozone exposition at the defective sites. *L. Valentini, I. Armentano, J. M. Kenny, C. Cantalini, L. Lozzi, and S. Santucci, Applied Physics Letters Volume 82, Issue 6, pp. 961-963 (2003); B. Delley, J. Chem. Phys. 113, 7756 (2000); ibid. 92(1), 508 (1990); D.B. Mawhinney, V. Naumenko, A. Kuznetsova and J.T. Yates Jr., J. Am. Chem. Soc., 122, 2383 (2000).*

NT-WeP20 Molecular Dynamics Study on the Non-equilibrium Flow of Small Molecules through Opened Carbon Nanotubes, K. Lee, S.B. Sinnott, University of Florida

Ultrafiltration membranes made of short, opened carbon nanotubes, which have relatively uniform nanometer-scale pore sizes and linear structures, may allow gases to selectively pass through the membrane. This potential

selectivity can be predicted from atomistic simulations of the diffusion and adsorption of the gases into and within carbon nanotubes. The computational nanofluidics of hydrocarbons, oxygen, and carbon dioxide has been studied with molecular dynamics simulations in the work reported here. The microscopic behaviors of these gases can be simulated with multiple integrations of the interactions among the atoms in a system. The transport of gas molecules for long time periods is characterized by initial non-equilibrium states followed by equilibrium states. The non-equilibrium state is induced by the diffusive motion of the gas molecules from one end of the nanotubes into the vacuum or low-pressure region at the other end of the nanotubes, and lasts until the gases are evenly distributed in the nanotubes. During the non-equilibrium state, the gas molecules move back and forth through the nanotubes. It is found that this behavior, the time needed for the attainment of equilibrium, and the molecular motions at the openings of the nanotubes are affected by the density (or pressure) of gas molecules both inside and outside of the carbon nanotubes. When the gas molecules reach the end of the nanotubes, the attractive force between the tube end and the gas molecules prevent the molecules from leaving the nanotube. In order to leave the tube, the molecules must be acted on by a repulsive force, which is exerted by other gas molecules entering at the other end. The dynamics of these various nonequilibrium diffusion regions are characterized and will be discussed in detail. In addition, a discussion of how the results change with changes in nanotube chirality and diameter will be discussed. This work is supported by the NASA Ames Research Center.

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