Poster Session 3

S1

Single-walled carbon nanotube ink for large area transparent conducting films by rod-coating method

Yan Meng, Xiao-Bing Xu, Hu Li, Yan Wang, Er-Xiong Ding, Ze-Chen Zhang, Hong-Zhang Geng

School of Material Science and Engineering, Tianjin Polytechnic University, Tianjin, Tianjin, China
genghz@tjpu.edu.cn

High concentration SWCNT ink was obtained by dispersing single-walled carbon nanotubes (SWCNTs) in water with the help of a combination of surfactants. SWCNT transparent conducting films (TCFs) were fabricated by rod-coating using the SWCNT ink. The combination of two surfactants provided optimal rheological behaviour, which produced uniform films by preventing dewetting and rupture of SWCNTs during drying. The combination led to a dramatic increase of shear viscosities but no change of their wettability. The viscosity of SWCNT ink was controlled by the ratio of two surfactants. The thickness of SWCNT films could be easily varied by controlling both the concentration of SWCNT ink and the size of the wire-wound rod. The TCFs were characterized using scanning electron microscope, transmission electron microscope, and Raman spectroscopy. The produced uniform SWCNT-TCFs treated by nitric acid have a relatively low sheet resistance with high transmittance. The performance has a wide range of applied interest for touch screen and flexible electronics.

S2

A simple method for purifying metallic impurities from Multi-walled Carbon Nanotubes by Chloroform Treatment

Chang Jin Lim, Naesung Lee, Jeung Choon Goak, Elsye Agustina

Nano engineering, Sejong univeristy, Seoul, South Korea, Korea, Republic Of
changjinlim@naver.com

INTRODUCTION

Carbon nanotubes (CNTs) renowned of their excellent properties, such as mechanical strength, chemical stability, as well as the high thermal and electrical conductivity, have encouraged the researches to actively evolve the application of CNTs in various industries. [1] However, the as-prepared CNTs are generally accompanied by carbonaceous or metallic impurities due to the utilization of metallic catalyst during the synthesis process.

Despite of the easily removed carbonaceous impurities, the encapsulated metallic impurities are chemically and thermally stable which prevent their removal due to the exposing difficulties. In comparison to single-walled carbon nanotubes (SWCNTs), multi-walled carbon nanotubes (MWCNTs) have less carbonaceous impurities, whilst on the other hand have more hardly removed metallic impurities.

The metallic impurities of MWCNT are serious impediments of the higher value-added applications (ex: lithium-ion batteries, high voltage cable) due to decrement of thermal stability, electrical and magnetic properties .Therefore, metal impurities should be removed, in which an effective purification technology is needed.[2]

Up to now, various CNT purification methods have been developed such as liquid phase oxidation by strong acid [3], microwave-assisted-method [4], or vacuum-high temperature treatment[5]. In this study, we developed an effective purification method in terms of time, cost and environmental friendly for overcoming problems of conventional methods.

MATERIALS AND METHODS

Material

MWCNT used in this study was synthesized by chemical vapor deposition method using iron (Fe) and cobalt (Co) catalyst on alumina (Al_2O_3) support from JEIO.

Chloroform is anhydrous from SigmaAldrich.

Method

CNT was placed in a glass tube located in the vertical-furnace at the nitrogen atmosphere and heated from room temperature to 500~900℃. When each of set temperatures is reached, chloroform carried by nitrogen gas was injected for one hour by 100mL/min flow rate allowing the reaction to take place. After 1 hour purification at the assigned temperature, furnace was cooled down to room temperature at the nitrogen atmosphere.

Nitrogen as the common industrially carrier gas was used to vaporize chloroform reagent which then injected to pass through the CNTs. Without strong acid or complex treatment, metal

impurities can be easily removed by this purification method. It is noteworthy that a simple cold trap can be applied to secure the by-product of reaction, such as the excessive chloroform and hydrochloric acid, preventing the hazardous chemical to escape. Purification occurs via chloroform decomposition to generate HCl gas and Cl radical which react with the metallic impurities in CNT. At the high temperature applied, metal chlorides in gaseous state ($FeCl_3$, $CoCl_2$, $AlCl_3$) were produced.and directly emitted through exit hood.

RESULTS AND DISCUSSION

According to the analysis of ICP-AES for the purified CNT, concentration of metallic impurities decreases linearly along with the increment of the reaction temperature from 500 to 800℃ while above 800℃, effect is saturated. Unpurified CNT contained 14,670 ppm of metallic impurities (Fe, Co, Al) and reduced to 535ppm after purification achieving 96.4% elimination.

EDX spectra indicate the change of surface state of the sample after treatment with chloroform. The contents of aluminium, iron, cobalt and oxygen is decreased while that of chlorine increased. Chlorine radical can be attached to the end of CNTs forming functional group chlorine containing instead of oxygen containing.

Raman spectroscopy shows that the crystalline quality is preserved or improved slightly during chloroform treatment.

CONCLUSIONS

In this work, chloroform treatment is proposed to eliminate metal impurities and advanced some properties such as thermal stability, crystalline quality. This system was optimized at 800℃ for 1 h, where the purification efficiency is ~96.4%, producing MWCNTs as pure as 99.95% (metallic impurities ~ 524 ppm). During purification, chlorine is functionalized to the surface of CNT, it changes surface properties of CNT. This can be detected by EDX On the Raman analysis, at a higher purification temperature, IG/ID was slightly improved, indicating that CNTs became more highly crystalline. On the TGA analysis, at a higher purification temperature, the oxidation temperature became higher, probably because a larger amount of metallic impurities were removed at a higher temperature. This purification process is simple, environmentally friendly, and cheap, so that our method is expected to be applied to preparation of highly pure MWCNTs for Li-ion batteries, supercapacitors, HV cables, etc.

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S3

Substituent Effect of Small Aromatic Solubilizers on Selective Separation of Single-Walled Carbon Nanotubes

Masashi Fukuzawa, Fumiyuki Toshimitsu, Yuichi Kato, Naotoshi Nakashima

Dept. of Applied Chemistry, Kyushu University, Fukuoka, Fukuoka, Japan
m.fukuzawa@mail.cstm.kyushu-u.ac.jp

Single-walled carbon nanotubes (SWNTs) are classified into metallic- or semiconducting-SWNTs according to their chirality. They are produced as a mixture of various chiralities. It would be a great break through to develop an efficient method of extracting specific chiralities of the SWNTs.

We previously revealed that the flavin with alkyl chains can selectively solubilize/extract semiconducting-SWNTs. Here we report a substituent effect of the flavin on the selective separation of the SWNTs by changing alkyl chains. The effects were investigated by several spectroscopies, which revealed the supramolecular interactions of flavins are important.

S4

Structure separation of metallic SWCNTs using gel column chromatography

Takeshi Tanaka, Yasuko Urabe, Takuya Hirakawa, Hiromichi Kataura

Nanosystem Research Institute, National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Ibaraki, Japan
tanaka-t@aist.go.jp

As-produced single-wall carbon nanotubes (SWCNTs) always contain various structures (chiralities). Recently we reported single chirality separation of semiconducting (S-) SWCNTs using gel column chromatography [1,2]. In this separation, metallic (M-) SWCNTs were not adsorb to the gel and not separated from each other. Here we report chirality separation of M-SWCNTs.Proceeding chirality separation of M-SWCNTs, M/S separation of HiPco-SWCNTs was conducted. The separated M-SWCNTs mixture was concentrated and applied to 60 cm long column packed with Sephacryl S-200 gel equilibrated with 0.3% SDS. After flowing 0.3% SDS, the concentration of elution solution changed to 0.4%, 0.5% SDS and 2% sodium cholate. From the optical adsorption spectra of eluted fractions, clear difference of adsorption peaks was observed, indicating chirality separation of M-SWCNTs. The early and late eluted fractions tended to contain M-SWCNTs with optical absorption peaks of longer and shorter wavelengths, respectively. Detailed analyses containing the result of Raman spectroscopy will be presented.

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S5

Full-length selective removal of metallic single-walled carbon nanotubes by organic film-assisted electrical breakdown

Keigo Otsuka, Taiki Inoue, Shohei Chiashi, Shigeo Maruyama

Department of Mechanical Engineering, The University of Tokyo, Bunkyo-ku, Tokyo, Japan
otsuka@photon.t.u-tokyo.ac.jp

Horizontal arrays of semiconducting (s-) single walled carbon nanotubes (SWNTs) grown on crystal quartz substrates have potential for the use in field effect transistor channels, especially for the SWNT-based large-scale integrated circuits. However metallic (m-) SWNTs simultaneously grown in the SWNT arrays are one of the major obstacles to the realization of high-performance devices. Although many researchers have been tackling on this problem, satisfying the required purity of s-SWNTs and purification scalability is still of great difficulty.

Here we present an organic film-assisted electrical breakdown method, which creates the horizontal arrays of pure s-SWNTs by removing full length of metallic SWNTs. SWNT arrays grown on quartz substrates were transferred onto Si/SiO_2 substrates for control of the electrical conductivity of s-SWNTs. Ti/Pd electrodes were patterned for voltage application on the SWNT arrays in their axial direction, followed by organic film deposition on the SWNT arrays. Performing electrical breakdown on the SWNT arrays embedded in the films resulted in over 100 times as long removal of m-SWNTs as conventional electrical breakdown technique. Remaining s-SWNT arrays after the breakdown can be used for the fabrication of the SWNT-based integrated circuits.

S6

Effect of Temperature on The Selection of Semiconducting Single Walled Carbon Nanotubes using Poly(3-dodecylthiophene-2,5-diyl)

Widianta Gomulya, Jorge Mario Salazar Rios, Vladimir Derenskyi, Stefan Jung, Satria Zulkarnaen Bisri, Sybille Allard, Ullrich Scherf, Maria Antonietta Loi

Physics, Zernike Institute for Advanced Materials, University of Groningen, Groningen, Groningen, Netherlands
Chemistry, Institute for Polymer Technology, Wuppertal University, Wuppertal, North Rhine-Westphalia, Germany
Physics, Zernike Institute for Advanced Materials, University of Groningen, Groningen, Groningen, Netherlands
Chemistry, Institute for Polymer Technology, Wuppertal University, Wuppertal, North Rhine-Westphalia, Germany
Physics, Zernike Institute for Advanced Materials, University of Groningen, Groningen, Groningen, Netherlands
w.gomulya@rug.nl

Polymer wrapped single-walled carbon nanotube (SWNT) has been demonstrated to be a very efficient technique to obtain high purity semiconducting SWNT. To obtain not only the high quality but also high concentration of SWNTs, a careful control of the physical parameter during the selection process is indispensable. Here, we report on the investigation of the influence of temperature on the selective dispersion of SWNT by Poly(3-dodecylthiophene-2,5-diyl) (P3DDT) wrapping. The interaction mechanism between the polymer chains and the semiconducting SWNTs are studied by controlling the temperature before, during, and after the selection process. Optical absorption and photoluminescence measurements are used to study the degree of interaction between the polymer in different aggregation forms and the carbon nanotubes. Better SWNTs individualization is achieved when the polymer is completely solubilized before the sonication process. The highest concentration of SWNT in the dispersion is obtained when the sonication is performed at low temperature. The high quality of the sample obtained with fully solubilized polymer is confirmed by the fabrication of field effect transistors, which demonstrate higher on-current and higher carrier mobility (up to 1.5 times) than those fabricated from SWNT solutions wrapped with polymer solubilized at low temperature.

S7

The effect of DNA adsorption on optical transition in mono-disperesed single-walled carbon nanotube

Masahiro Ito, Yusuke Ito, Daisuke Nii, Hiroki Kato, Kazuo Umemura, Yoshikazu Homma

Department of physics, Tokyo University of Science, Shinjuku, Tokyo, Japan
m-ito@rs.tus.ac.jp

Single-walled carbon nanotubes (SWNTs) are solubilized and functionalized by means of DNA adsorption onto the surface [1,2]. The hybridization of DNA and SWNT (DNA-SWNT) is expected to have applications in chemical and biochemical fields. As a result of DNA-SWNT dispersed in solution, photoluminescence (PL) can be observed from SWNTs. PL from an SWNT is sensitive to the surrounding environment, and should reflect the impact of adsorbed DNA on SWNT. However PL from DNA-SWNT ensemble makes spectrum analysis difficult. Recently, we revealed that PL could be obtained even from DNA-SWNT attaching to a substrate under the dry condition [3]. In this study, we investigated the intrinsic effect of single stranded DNA (ssDNA) and double stranded DNA (dsDNA) adsorption on the optical transition in mono-dispersed DNA-SWNTs under the dry condition.

The DNA-SWNT solution was diluted low enough with pure water. We obtained PL spectra from individual DNA-SWNTs on the substrate. We compared PL spectra from the same chirality SWNT under five different conditions: SWNT suspended between micro structures in air, ssDNA-SWNT in solution, ssDNA –SWNT under the dry condition, dsDNA-SWNT in solution and dsDNA-SWNT under the dry condition. The optical transition wavelength showed clear red shifts in the order, suspended SWNT, ssDNA-SWNT and dsDNA-SWNT. Furthermore, the emission wavelength from ssDNA-SWNT was different between under the wet and dry condition, while the emission wavelength of dsDNA-SWNT was retained the same. The present results imply that excitonic transition is sensitively influenced by the local environment on the SWNT surface, such as the molecular configuration and density.

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S8

Thermodynamics of Quasi-Epitaxial Assembly of FMN around various (n,m)-SWNTs

Fotios Papadimitrakopoulos, Roholah Sharifi, Milinda Samaraweera, José A. Gascón

Chemistry, Institute of Materials Science, Storrs, CT, United States
papadim@ims.uconn.edu

The helical assembly of self-organized flavin mononucleotide (FMN) around single-walled carbon nanotubes (SWNTs) presents among the highest organization order in nanotube surfactants. In this contribution, we quantitatively analyze and model for the first time the cooperative hydrogen bonding of adjacent flavin moieties as well as the concentric π–π interactions between the isoalloxazine moieties and the underlying graphene lattice as a function of (n,m)-SWNT chirality. For this we use dissociation thermodynamics of FMN-wrapped (n,m)-SWNTs dispersed in both H_2O and D_2O as a function of FMN concentration. The binding strength of these FMN assemblies has been assessed in terms of ΔH, ΔS and ΔG. Atomistic molecular simulations were used to modeled these data and link their dependence in terms of nanotube diameter (d_t) the chiral angle (ϕ). The findings of this study provide the first quantitative proof of the quasi-epitaxial assembly of FMN around various (n,m)-SWNTs. This study demonstrates the architectural fidelity of FMN-wrapped SWNTs that closely emulates the dissociation mechanics of double-stranded DNA in its aqueous solutions.

S9

In situ characterization of DGU separation by full 2D fluorescence-excitation and resonant Raman spectroscopy in the centrifuge tube

Sofie Cambré, Pieter Muyshondt, Remi Federicci, Wim Wenseleers

Physics department, University of Antwerp, Antwerp, Antwerp, Belgium
Wim.Wenseleers@uantwerp.be

Density gradient ultracentrifugation (DGU) is one of the most promising techniques for chirality sorting of carbon nanotubes (CNTs),[1] but the separation mechanisms are not quite fully understood, as they depend on the subtle differences in surfactant coating and filling[2,3] of CNTs. Proper characterization of composition and filling requires dedicated spectroscopic techniques, not available in analytical centrifuges. We present ultrasensitive setups capable of recording full 2D IR fluorescence-excitation maps and high resolution resonant Raman spectra at various laser wavelengths, in situ in the centrifuge tube as a function of position (density) immediately after DGU separation. Applied to isopycnic DGU, this yields far more precise information on the density of surfactant coated SWNTs as a function of their diameter and chiral structure, than achievable by studying extracted aliquots. This in turn yields useful information on the diameter-dependent surfactant coating, which is also crucial to the understanding of other separation methods, such as aqueous two-phase extraction [4,5]. The variation in surfactant stacking explains for instance the long standing paradox that sodium cholate would provide a better diameter sorting, while the extremely similar deoxycholate is the more efficient surfactant for solubilizing the nanotubes.

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S10
The functionalization of multi-walled carbon nanotubes and carbon materials for application in gas separation and gas sensors

John A. A. Gibson, Andrei V. Gromov, Ben Hunter, Stefano Brandani, Eleanor E. B. Campbell

School of Engineering and School of Chemistry, University of Edinburgh, Edinburgh, Scotland, United Kingdom
School of Chemistry, University of Edinburgh, Edinburgh, Scotland, United Kingdom
School of Engineering, University of Edinburgh, Edinburgh, Scotland, United Kingdom
School of Chemistry, University of Edinburgh, Edinburgh, Scotland, United Kingdom
A.Gibson@ed.ac.uk

Multi-walled carbon nanotubes (MWCNTs) can be functionalized to enhance their mechanical, electrical and thermal properties for a given application. Functionalized MWCNTs have potential applications ranging from gas sensors to catalysis.

Amine moieties were grafted to the surface of the MWCNTs by both a one-step and two-step functionalization process. In the one-step process lithium, ethylene diamine (EDA) and MWCNTs were reacted to directly graft EDA to the surface. In the two step process, the nanotubes were first oxidised by a variety of techniques, including air plasma and reaction with H_2SO_4/H_2O_2. Second, the oxygen groups formed on the surface were used to covalently graft derivatives of polyethylenimine to the surface. The extent and success of the functionalization was studied by IR, XPS, laser ablation mass spectrometry and gas adsorption analysis.

Amino functionalized carbon nanotubes and carbon materials have potential application in carbon capture, through the adsorption based gas separation of CO_2 from the flu gas of fossil fuelled powered power stations. The basic amino group provides chemisorption-based selectivity for the slightly acidic CO_2 gas with respect to the non-acidic components of the flue gas mixture. The CO_2 uptake of the adsorbents was investigated by thermal gravimetric analysis and by the zero length column experiment. The effect of the different weight loadings of amine, on porous activated carbon, on the available pore volume, surface area and the change in the pore size distribution was investigated by nitrogen adsorption analysis. An increase in the CO_2 capacity was observed with the introduction of amine groups. The heat of adsorption was investigated in an attempt to understand the extent and mechanism of the chemisorption.

To investigate the electrical properties of the functionalized MWCNTs, thin films, or bucky papers, were prepared. The sheet resistivity of the various functionalised films was measured by the van der Pauw method under various gas atmospheres, (N_2, O_2 and CO_2).We report a change in the sheet resistivity on exposure to the different atmospheres.

S11

Single chirality desorption of single-wall carbon nanotubes using mixed surfactant gel chromatography

Yohei Yomogida, Mayumi Tsuzuki, Atsushi Hirano, Shunjiro Fujii, Takeshi Tanaka, Hiromichi Kataura

Nanosystem Research Institute, National Institute of Advanced Industrial Science and Technology, Tsukuba, Ibaraki, Japan
y-yomogida@aist.go.jp

Previously, we reported a multicolumn gel chromatography method that can separate single chirality single-wall carbon nanotubes (SWCNTs), where a chirality selective adsorption of SWCNT to the gel was utilized [1]. In this study, we have improved the elution process of a single column chromatography and have demonstrated a single chirality separation using chirality selective desorption of SWCNTs.

We used aqueous solution of mixed surfactants, sodium dodecyl sulfate (SDS) and sodium deoxycholate (DOC) for the chirality selective elution. Precisely controlled DOC concentration enabled chirality selective elution and high-purity (6,5) and (7,6) SWCNTs were obtained from a tiny amount of HiPco solution that was not sufficient for the multicolumn method. The chirality distribution of each fraction was analyzed in detail using optical absorption and photoluminescence (PL) spectra. Results suggest the desorption process is highly diameter selective probably due to diameter selective DOC adsorption to SWCNTs. Because this separation is based on a quite simple adsorption chromatography, this method can be easily applicable to a large scale separation of single-chirality SWCNTs.

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S12
Chirality sorting of SWCNTs using gradient elution in gel column chromatography

Hiromichi Kataura, Mayumi Tsuzuki, Takeshi Tanaka

Nanosystem Research Institute, National Institute of Advanced Industrial Science and Technology, Tsukuba, Ibaraki, Japan
h-kataura@aist.go.jp

Because single-wall carbon nanotube (SWCNT) has three different C-C bonds due to its tiny cylindrical structure, to know the precise structure of SWCNT is very important for understanding its physical properties. For this purpose, large scale separation of single-chirality SWCNT is one of the most important research targets, which enables growing a single crystal for X-ray diffraction measurement. Previously, we reported that diameter selective desorption of SWCNTs was possible using different concentration of sodium deoxycholate (DOC) in the gel column chromatography [1]. In this work, we have extended this method into mixed surfactant system and tried a gradient elution for DOC concentration using a high performance liquid chromatography (HPLC). In this system, we can get UV-VIS-NIR absorption spectra in situ, which showed clear chirality sorting for HiPco SWCNTs. Because "overloading effect" is not necessary in this method, this is more effective for large scale separation of single-chirality SWCNTs than the multicolumn method that we reported previously [2].
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S13

Structure and sorting of DWNTs by diameter and helicity

Annick Loiseau, Ahmed Ghedjatti, Frederic Fossard, Emmanuel Flahaut, Jean-Sébastien Lauret

LEM, ONERA-CNRS, Chatillon, France, France
CIRIMAT, Université de Toulouse III, Toulouse, France, France
LAC, ENS Cachan, Cachan, Cachan, France
loiseau@onera.fr

Single wall carbon nanotubes (SWCNTs) have shown extrordinary electronic and optical properties since their discovery in 1991 by Iijima et al [1]. However, these properties are highly sensitive to any slight fluctuation in their environment, making their control difficult. On the contrary, double wall nanotubes (DWCNT) offer the advantage that the outer layer can be used as a sacrificial layer, preventing any degradation of the electronic structure of the inner layer [2]. However, interaction between the layers and its impact on their respective properties require to be known in detail.

This is the purpose of the study we have undertaken. DWCNT have been elaborated using CVD techniques [3] In this process, CH_4 is thermally decomposed on Co:Mo-MgO catalysts, in order to reach a fraction of DWNTs close to 80%. As a first step, we performed a detailed analysis, by TEM, of the CNT population.

We have found a population of DWNT close to the forcast of 80%. We have observed that nanotubes with an homogeneous diameter distribution, are often grouped into bundles. Statistical analysis of the diameter distribution indicates that diameter of DWNTs down to 1.4 nm can be obtained.

Moreover, thanks to a Cs-corrected JEOL ARM200 operating at 80kV, we have been able to image directly the chiralities of either the intern or the extern wall for different tubes. Numeric FFT of the images have been compared to diffraction simulations using a Fortran code developped by Lambin and Kociak [4]. Results show an excellent agreement between measurements and simulations which confort us in the reliability of this study.

In order to rely diameter distribution, chirality and optical properties, we sorted the primary solution, using the density gradient ultracentrifugation (DGU) and perform optical absorption measurements and TEM for different populations of CNTs. We present our first results on optical and structural characterization of sorted DWCNTs.

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